

## Bio-sorption of Copper, Nickel, Iron and Zinc onto *Carica papaya* L. leaves: mechanism and operating parameters on the adsorption yield.

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**Abstract:** The aim of this study is to assess the ability of *Carica papaya* leaves to remove heavy metals from contaminated water. Batch experiments models were performed to investigate the optimal parameters such as the time contact, the adsorbent dose, the initial metal concentration. With the variation of concentration, the isotherms and kinetics adsorption were also evaluated. Ten minutes are sufficient to reach the maximum of 84.32% removal of iron from the water. Twenty minutes are necessary for reaching the maximum removal of nickel (24.01%), of copper (29.77 %), of zinc (16.40 %). A mass of 0.3 g of adsorbent is sufficient to reach the maximum removal of iron from the water while 0.5 g is required for the other three metals used in this study. The last parameters studied is the effect of the initial metal concentration. The maximum adsorption value is obtained for the initial concentration of 2.5 ppm for copper and 10 ppm for the three other metals. For copper and iron, the adsorption parameters are in good agreement with Tempkin isotherm. For Nickel and zinc the studied models are not suitable to explain their experimental isotherms. For all metals, the kinetics of the adsorption process are in accordance with the pseudo-second order model. Experimental values of the adsorption capacity are close proximity to the optimum values predicted by the pseudo-second order model.

**Keywords:** *Carica*, Adsorbent, Metal, Isotherm, Kinetic

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### I. Introduction

Environmental protection is a major subject of interest for both scientists and the public. More and more public authorities and citizens' organizations act to curb the extent of the problem of pollution of our soils and water resources by heavy metals. In soils and water, we found mainly at high levels the most toxic compounds such as lead, chromium, zinc, copper, nickel, arsenic, cadmium, mercury, silver and uranium. The danger of these compounds is due to their high remanence in the environment. The cost for the elimination of these compounds in soil and water by classical physico-chemical methods such as chemical precipitation [1], recovery by electrolysis [2], the use of ion exchange resins [3], solvent extraction [4] or membrane filtration [5] is very high. These methods push the industrialists who are the main polluter to neglect this problem. Indeed, these methods are excessively expensive and give only low yields for metal concentrations below 100 ppm [6]. Besides, these methods generate large amounts of sludge after treatment. In developing countries, small industrial cannot implement these conventional depollution processes. Therefore, it is urgent to develop alternative technologies for the elimination or reduction of heavy metals levels in the environment at lower costs.

Materials considered as agricultural, urban, and/or industrial waste having no financial interest can be used in phytoremediation or bioremediation. The results, concerning these methods, recorded in recent years [7], [8] are promising. The mobility of heavy metals in aquatic ecosystems and their high toxicities make these ions major pollutants. They cannot be destroyed by biological systems but can be oxidized or reduced. These redox reactions can change the solubility of the ion in water and make it bioavailable [9].

The ability of plants to accumulate heavy metals depends on several parameters such as the difference in plant species, the methods, conditions of cultivation of these plants. Depending on the pollutants, we can use some of the plants parts: leaves depending of their ages, roots, barks, etc,... Corn is a plant particularly suitable for accumulating lead mainly in its roots but also in its leaves and stem [10]. Some plants are the ability to render harmless soils pollutants by reducing their bioavailability. Indeed, these plants can immobilize some metals by transforming them into mineral compounds in the roots.

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Most of the agricultural by-products are widely available and they do not have any financial value. These by-products also pose a problem of storage and disposal. These materials can be used for pollution control or recovery of heavy metals in industrial flows [11]. After removal of pollutants, biomass can be easily regenerated and recycled [12], [13]. The origin and treatment of biomass also influence the quality of the pollution control and the quantity of pollutants removed. The sequestration of metals can involve several complex mechanisms, such as ion exchange, complexation, adsorption to surfaces by physical or chemical interactions. Several mechanisms are described for biosorption [14], [15].

It is in this context that we are trying to promote papaya leaves in the decontamination sector of water polluted by heavy metals.

## II. Material and Methods

### Preparation of the adsorbent

Red lady *Carica papaya* L. was harvested at the Sébikhotane protected forest region (14°43'14.4"N, 17°08'16.4"W). The leaves (CPL) were collected and rinsed twice with tap water first and distilled water to remove all the particles. They were shadow dried for three days. After this first process, the CPL were dried at 50°C for 48 hours. The CPL were ground using a grinder and sieved to have an average particle size of 0.25 mm. This CPL were stored in a plastic container.

### Batch adsorption tests

All the experiments were conducted in discontinuous batch. A weighed sample of CPL was mixed with 50 mL of the metal solution (copper, zinc, nickel and iron) in 150 mL conical flasks. The mixture was stirred for a fixed time at 25°C. After this process, the liquid was separated from the adsorbent by filtration through a Whatman Filter N°1. The residual metal concentration was determined using Atomic Absorption Spectrometer (ThermoFisher Scientific iCE 3000 Series). The experimental data were used to determine the removal capacity and the quantity of metal adsorbed on the CPL:

$$\text{Removal capacity} = \frac{(C_0 - C_e) \times V}{C_0} \times 100$$
$$q_e = \frac{(C_0 - C_e) \times V}{m}$$

Where  $C_0$  and  $C_e$  are respectively the initial and the final metal concentrations (mg/L) in the liquid phase,  $V$  (L) is the volume of the liquid phase and  $m$  (g) is the adsorbent mass used.

### Effect of the time contact on the removal capacity

The equilibrium time was determined using a concentrated metal solution of 10 mg/L. Each 50 mL metal solution was treated with 0.25 g of CPL at time ranging from 10 to 120 minutes. The experiments were conducted at the same pH and the flasks contents were stirred at 500 rpm at a temperature of 25°C.

### Effect of adsorbent dose on the removal capacity

50 mL of a 10 mg/L metal solution was treated with a mass of adsorbent ranging from 0.1 to 1.0 g. The experiments were conducted at the same pH and the flasks contents were shaken at 500 rpm at temperature of 25°C for the optimal time determined previously.

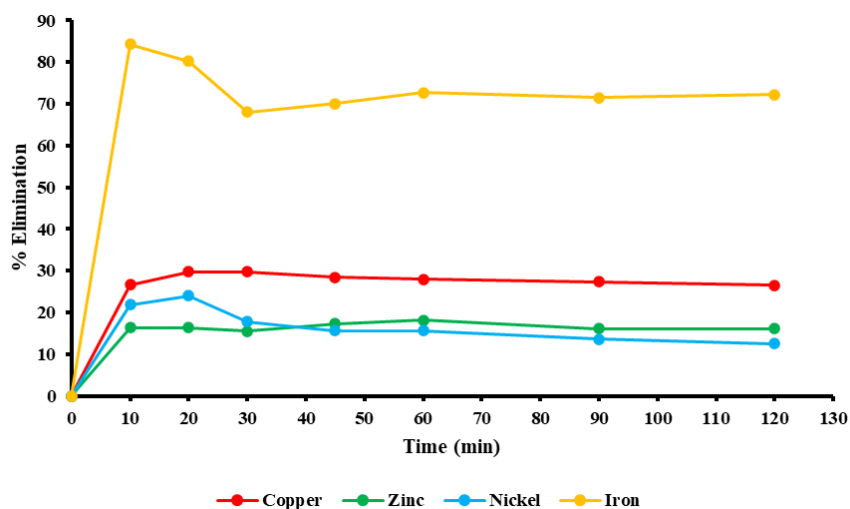
### Effect of metal concentration on the removal capacity

The effect of metal solution on the removal capacity were determined by setting the optimum temperature and adsorbent dose and varying the metal concentration between 2.5 and 20 mg/L. The flasks contents were stirred at 500 rpm at a temperature of 25°C.

## III. Results

### 3.1 Optimization of heavy metal removal parameters

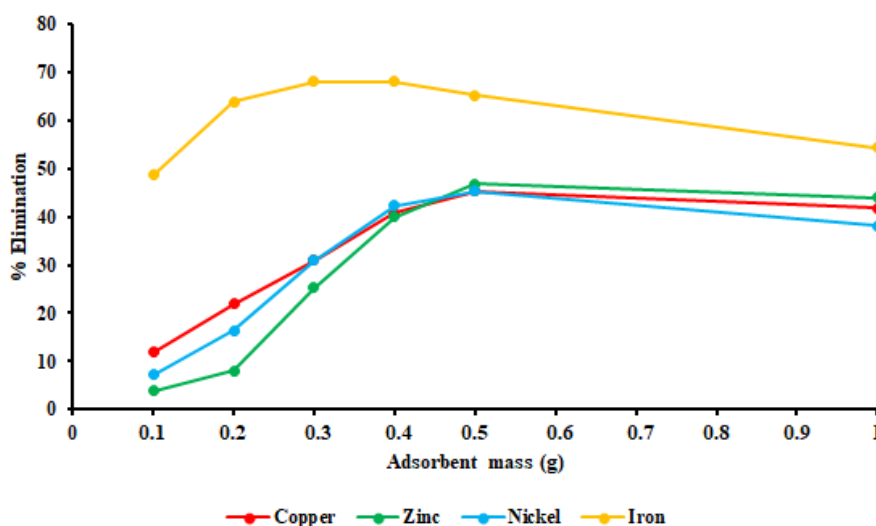
Figure 1 shows the removal percent of iron, nickel, copper, zinc over time. The dependence of metal adsorption over time is studied with an initial metal concentration of 10 ppm and a fixed mass of 250 mg of carica papaya leaves (CPL). For all the ions, a rapid increase in the rate of elimination is observed, reaching the maximum after 10 minutes for iron and 20 minutes for nickel, zinc, copper. For iron we have an elimination rate of 84.32 % after 10 minutes. When the time is extended, we observe a decrease in the elimination rate to 70% around 45 minutes. This rate remains stable up to 120 minutes of contact. For zinc the maximum is 16.40 % after 10 minutes and remains stable for up to 120 minutes. For nickel and copper, the maximum rate is 24% and 30% respectively after 20 minutes. These rates decrease and reach after 120 minutes 12.5% and 26% respectively for nickel and copper. It appears here that CPLs are more effective for removing iron.



**Figure 1:** Effect of contact time on the adsorption capacity of CPL

The influence of the dose of CPL on the rate of elimination of heavy metals in aqueous solutions was studied for masses ranging from 0.1 to 1 g with a pH = 7 and solutions of metals of 10 ppm of concentration. The rate of metal elimination is shown in figure 2. By increasing the dose of CPL from 2 to 20 g/L, we note that for iron the rate of elimination increases rapidly and reaches a maximum of 68% at a dose of 6 g/L. Between 6 g/L and 20 g/L, there is a release of the metal in the solution and the rate of elimination stabilizes at 54% for a dose of 20 g/L. The optimum dose for removing iron from the aqueous solution is 6 g/L. However, for nickel, copper and zinc the removal rate reaches a maximum of 10 g/L with 45% for nickel and copper and 47% for zinc. For these metals, the optimal dose is 10 g/L. Beyond this dose and up to 20 g/L, there is a slow decrease in the rate of elimination, which stabilizes at 42% for nickel, 38% for copper and 44% for zinc.

As expected, a high dose of adsorbent reduces saturation of adsorption sites. Chemical species are more shared due to the increased adsorbent surface area and the availability of adsorption sites. This is because the concentration of the adsorbate relative to the adsorbent drops and the system reaches equilibrium at lower removal rate values: the adsorption sites remain unsaturated. On the other hand, when the dose is low, chemical species can easily access and attach themselves to the adsorption sites which makes the elimination rate better with saturated adsorption sites. Similar behaviour has been observed in the case of removal of nickel, copper and zinc by *Moringa oleifera* [12] and in the case of removal of copper and lead by black tea [16].



**Figure 2:** Effect of adsorbent dose on the removal capacity

The adsorption capacity is strongly related to the initial concentration of the metal in solution. In Figure 3, we note that for copper, the adsorption capacity reaches its maximum for a concentration of 2.5 ppm and decreases sharply when the initial metal concentration is varied from 2.5 ppm to 20 ppm. For zinc and nickel, the adsorption is maximum at 10 ppm. However, a release phenomenon is observed between 2.5 and 5 ppm for

nickel and between 5 and 7.5 ppm for zinc. The maximum adsorption for iron is reached at 10 ppm. It is found that at a low concentration all the ions would reach specific adsorption sites, which facilitates the removal of the pollutant. However, at high doses, saturation of specific sites occurs, the exchange sites fill up. These exchange sites which are energetically less favourable for adsorption are involved in increasing the metal concentration of the solution. This observation has been reported several times in research works [17], [18]. For the iron, which is best removed by the adsorbent, it is noted that the adsorption capacity goes from 187.61 mg / g to 1668.8 mg / g when the initial concentration is increased from 2.5 ppm to 20 ppm. This may be due to a greater availability of iron ions in the solution. In addition, according to Basha et al. [18] a high concentration of ions in solution creates a driving force to overcome resistance to mass transfer from solution to adsorbent. The probability of collusion between the ions in solutions and the adsorbent is thus greatly increased. For the other three ions, there is also an increase in adsorption capacity from 55 mg / g to 103 mg / g for nickel, from 121 mg / g to 469 mg / g for copper and from 103 mg / g at 352 mg / g for zinc.

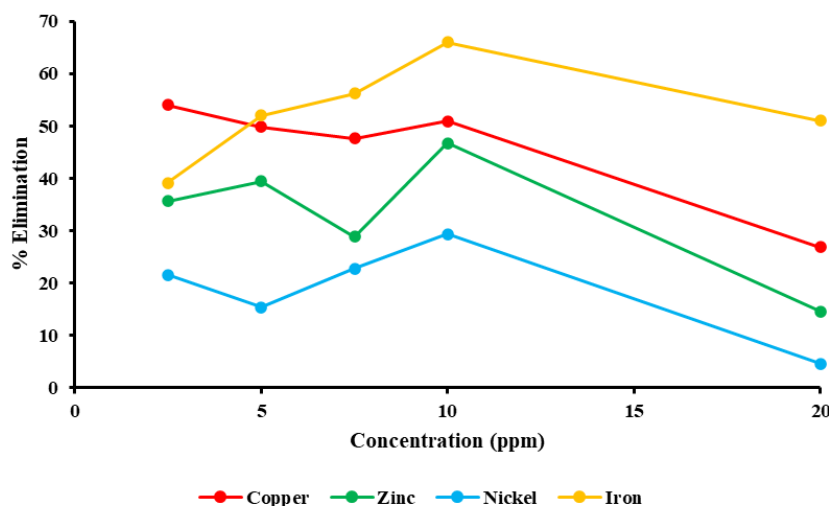


Figure 3: Effect of heavy metals initial concentration on the adsorption capacity of CPL

### 3.2 Adsorption isotherms

To understand how the adsorbed ions are distributed between the liquid phase and the solid phase consisting of the adsorbent, the adsorption isotherms are examined at equilibrium. The experimental data are analysed by fitting them with different models. To find the best model for design purposes, isotherm models named Langmuir, Freundlich, Temkin were used in this study [19].

The experimental equilibrium data for the adsorption of iron, copper, nickel, and zinc are correlated using the linear forms of the equations of Langmuir (1), Freundlich (2) and Temkin (3).

$$\frac{c_e}{q_e} = \frac{1}{K_L Q_0} + \frac{c_e}{Q_0} \quad (1)$$

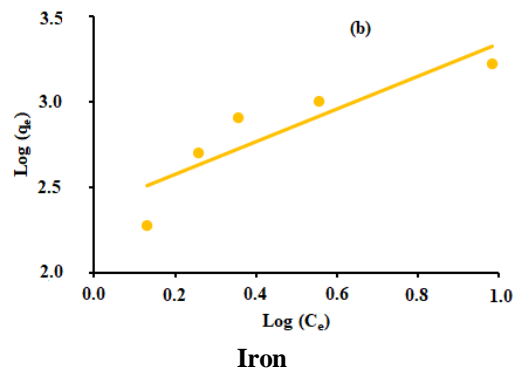
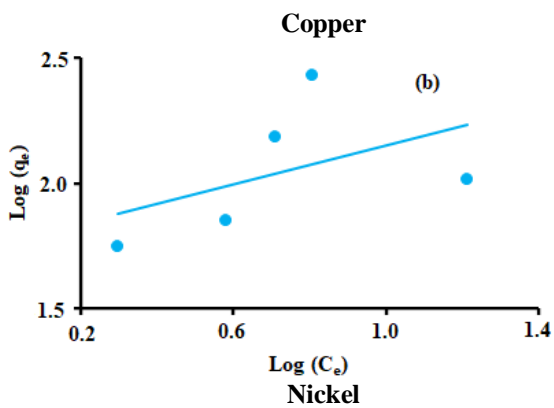
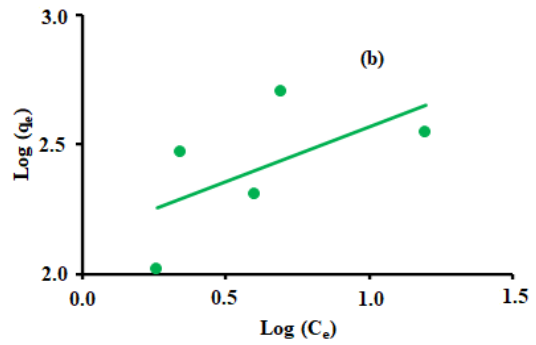
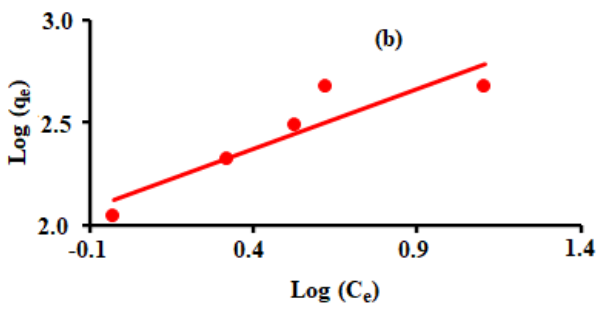
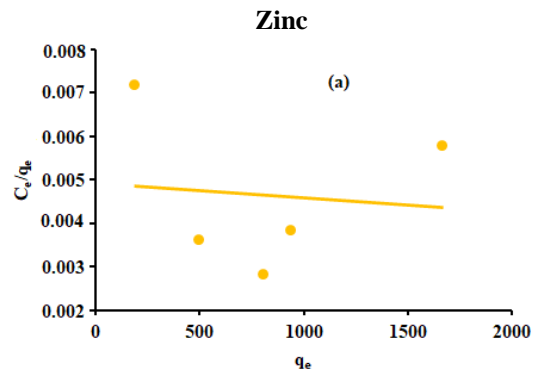
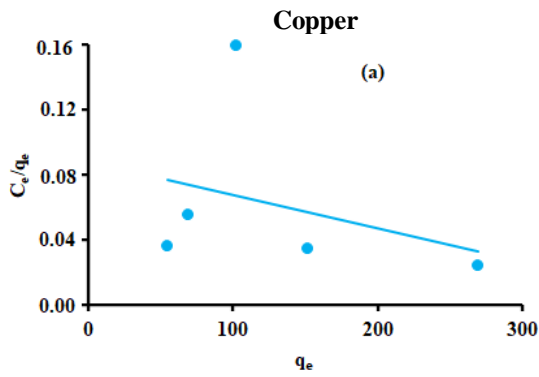
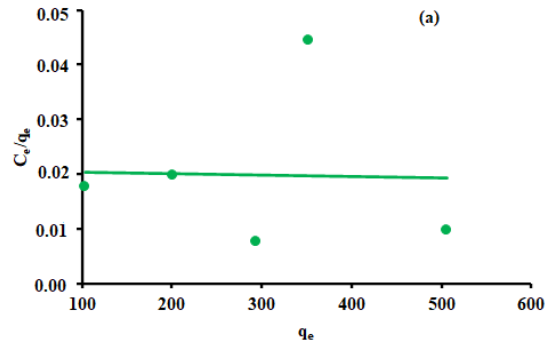
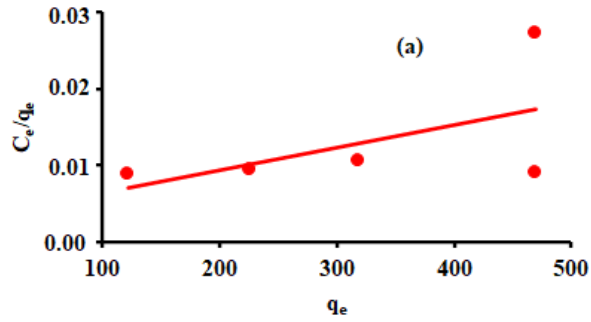
$$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e \quad (2)$$

$$q_e = \frac{RT}{b_t} \ln K_T + \frac{RT}{b_t} \ln C_e \quad (3)$$

Figure 4 represents the various linearized isotherms such as Langmuir, Freundlich and Temkin. The estimated parameters ( $Q_0$ ,  $K_L$ ,  $1/n$ ,  $K_F$ ,  $B$  and  $K_T$ ) of the set of isotherms are reported in Table 1. The values of the correlation coefficient of  $R^2$ , which are important to estimate the quality of the adjustment, are also shown in Table 1.

The iron and copper adsorption data correlate very well with Temkin's model with good correlation coefficients of 0.9949 and 0.9921 respectively, while the nickel and zinc adsorption data does not. are correlated with none of the three models studied (Table 1).

The data in Table 1 show that iron and copper follow the same pattern. Langmuir's model is the least suitable to match the experimental adsorption equilibrium data for the two metals. The correlation coefficient  $R^2$  is so low, with values of 0.01 for iron and 0.31 for copper. The experimental data of the two ions are not either, better adjusted by the Freundlich model which gives poor correlation coefficient of 0.79 and 0.82 respectively for iron and copper.



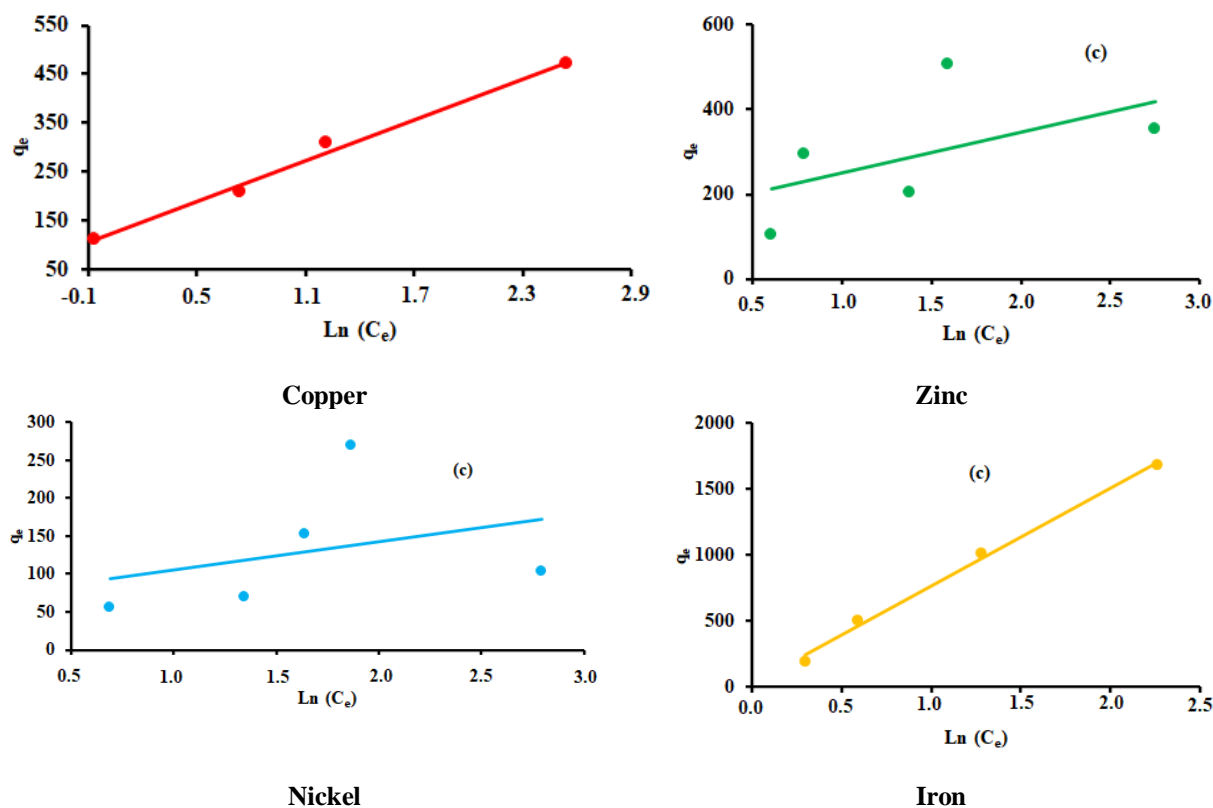


Figure 4: Metal adsorption isotherms : . Langmuir (a): Freundlich (b); Tempkin (c).

Table 1: Adsorption isotherms parameters

Metal	Langmuir			Freundlich			Temkin		
	$\frac{c_e}{q_e} = \frac{1}{K_L Q_0} + \frac{c_e}{Q_0}$			$\ln q_e = \ln K_F + \frac{1}{n} \ln C_e$			$q_e = \frac{RT}{b_t} \ln K_T + \frac{RT}{b_t} \ln C_e$		
	$Q_0$ (mg/g)	$K_L$ (L/mg)	$R^2$	$1/n$	$K_F$	$R^2$	$b_t$ (J/mol)	$K_T$ (L/mole)	$R^2$
<b>Copper</b>	16667	-0.0286	0.813	0.5452	131.04	0.937	17.76	2.34	0.992
<b>Zinc</b>	-333 333	-0.0001	0.001	0.4224	140.02	0.349	25.87	5.02	0.283
<b>Nickel</b>	-5000	-0.0023	0.103	0.3882	57.73	0.224	65.66	5.90	0.113
<b>Iron</b>	-3 333 333	-0.0001	0.010	0.9585	243.33	0.786	3.33	1.03	0.995

For these two ions, the Temkin model proved to be the best model for fitting the experimental data, showing excellent correlation coefficient values of 0.9948 and 0.9921 respectively for iron and copper. The values of the Temkin's isotherm constant  $b_t$  are 3.33 and 17.76  $\text{KJ mol}^{-1}$  respectively for iron and copper. It is known that the  $b_t$  value in the range 8 – 16  $\text{KJ mol}^{-1}$  indicates an excellent verification of the Temkin model [20]. These values indicate strong interactions between iron and the adsorbent, whereas between copper and the adsorbent there are weak interactions, supporting an ion exchange mechanism. This suggests that there is a uniform distribution of binding energy over the population of adsorption sites, thus supporting a homogeneous adsorption mechanism.

#### Adsorption kinetics

Kinetic studies of the adsorption of metal ions, iron, nickel, copper and zinc on CPL were carried out at pH = 7, at a temperature of 25°C and at a fixed adsorbent concentration of 6 g/L for iron and 10 g/L for nickel, copper and zinc. Different initial concentrations of metal were used (2.5 ppm to 20 ppm). Aqueous metal

concentrations were measured in a similar fashion, as a function of contact time. The amount of  $q_t$  (mg/g) adsorbed by the adsorbent at time  $t$  is calculated using Equation 4:

$$q_t = \frac{(C_0 - C_t)V}{m_0} \quad (4)$$

Four kinetic models: pseudo-first order, pseudo-second order, intra-particle diffusion model, Elovich model (Table 2) were examined to understand the adsorption mechanism on CPL. The suitability of each model was verified by correlating the parameters of the linear regression equation on the data and values provided experimentally by using four different equations.

In order to study the adsorption kinetics of  $Fe^{2+}$ ,  $Ni^{2+}$ ,  $Cu^{2+}$  and  $Zn^{2+}$  ions on CPL, four different kinetic models are used to correlate the data obtained from the elimination experiments of these ions. These are pseudo-first order (5), pseudo-second order (6), intraparticle diffusion (7) and Elovich (8):

$$\ln(q_e - q_t) = \ln q_e - k_1 t \quad (5)$$

$$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{t}{q_e} \quad (6)$$

$$q_t = k_i t^2 + I \quad (7)$$

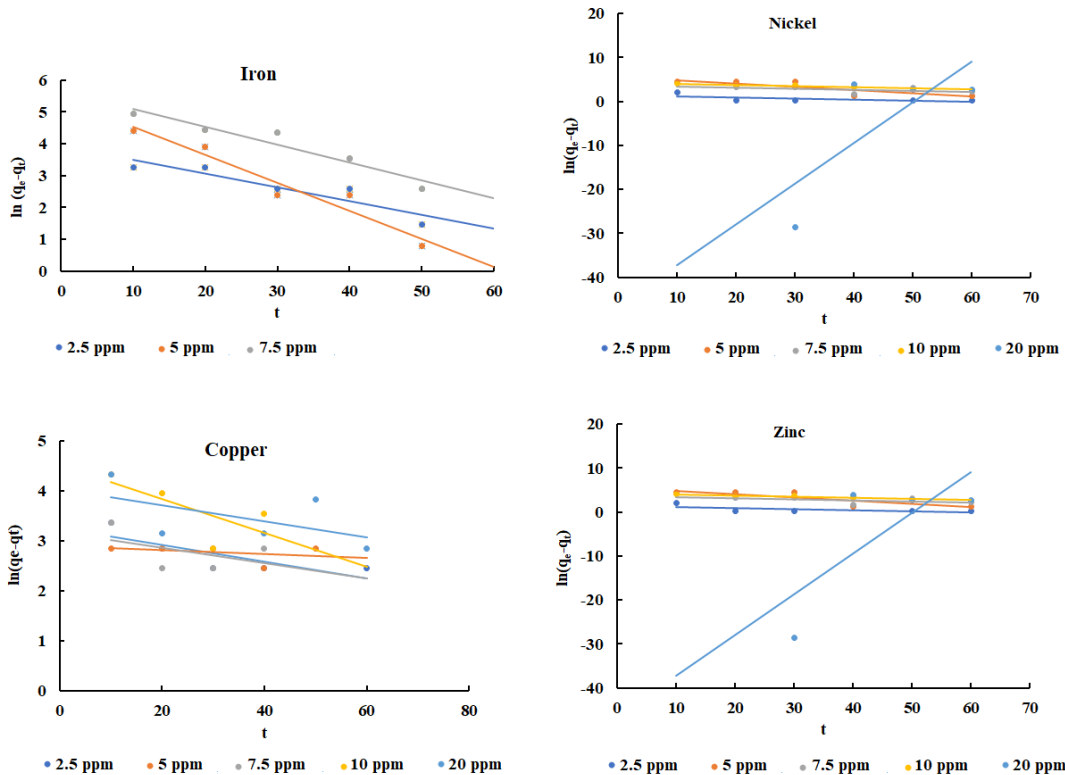
$$q_t = \frac{1}{\beta} \ln(\alpha\beta) + \frac{1}{\beta} \ln t \quad (8)$$

Table 2 collates the parameters obtained from the results of adsorption kinetics studies.

When using the pseudo-first order model equation, the results of  $q_{e,cal}$  obtained for all the ions studied diverge from the experimental values of  $q_{e,exp}$  significantly. On top of that, all the correlation coefficients are much lower than unity (Table 2). These observations clearly indicate that the pseudo-first order model does not describe any of the adsorption kinetics of iron, nickel, copper, or zinc. When we use the pseudo-second order model equation, we observe a good correlation of the data by this model. For all the ions studied and for all the initial metal concentrations, the correlation coefficients are close to unity when using the kinetic equation of the pseudo-second order model (Figure 5). The calculated  $q_{e,cal}$  values also agree with the experimental data in the case of pseudo-second order kinetics with small deviations (Figure 5, Table 2). These observations indicate that the adsorption data are well represented by pseudo-second order kinetics in the case of iron, nickel, copper, and zinc ions. It is also observed that the value of the adsorption rate constant  $k_2$  decreases with the increase of the initial metal concentration. This behaviour can be attributed to the weak competition for surface adsorption sites at a lower concentration. For high copper concentrations, the strong competition of active surface sites and the repulsion between the adsorbed ions and the ions in solution results in low adsorption rates. Similar behaviours have been observed in the adsorption of certain transition metals to other adsorbent materials from biomass [15], [17], [21]. Other kinetic models give exceptionally low values for the correlation coefficient and cannot describe the mode of absorption of iron and copper. Indeed, the correlation coefficients resulting from the pseudo-first order model are very weak (less than 0.80) and very large deviations are observed between  $q_{e,cal}$  (calculated) and  $q_{e,exp}$  (experimental) values (Table 2). This indicates that the adsorption of these ions does not follow pseudo-first order kinetics (Figure 5, Table 2). The correlation coefficients in the case of the intra-particle diffusion model (Figure 5) and the Elovich model (Figure 5) are also extremely far from unity. Indeed, we observe in figure 5 that for all metals, the relationship between the adsorbed quantity and the square root of time is not linear, thus suggesting that the intra-particle diffusion is not the kinetics which governs the adsorption. Figure 5 shows that for all metals, the relationship between the amount adsorbed and the logarithm of time is not linear. It emerges from these findings that the adsorption kinetics by CPL of  $Fe^{2+}$ ,  $Ni^{2+}$ ,  $Cu^{2+}$  and  $Zn^{2+}$  ions are exclusively described by the pseudo-second order model and that only one adsorption mode takes place during this process.

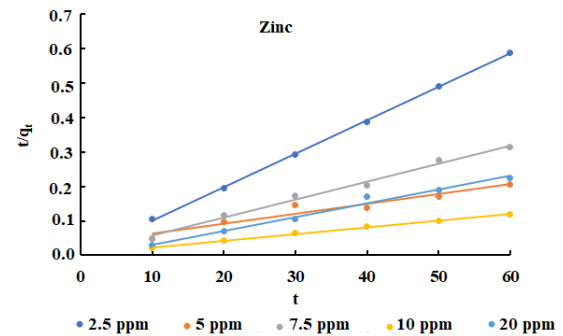
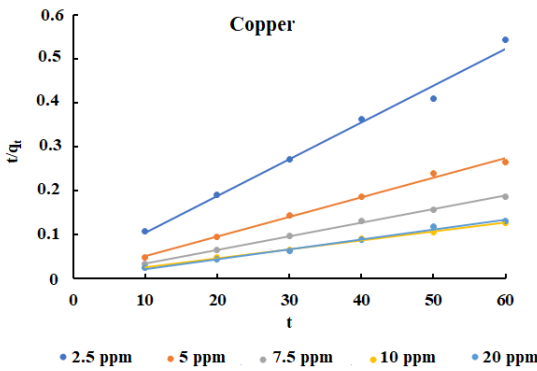
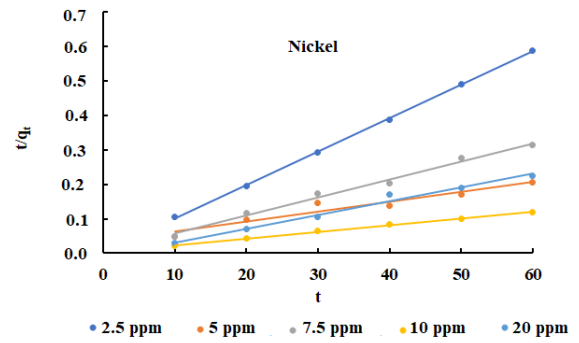
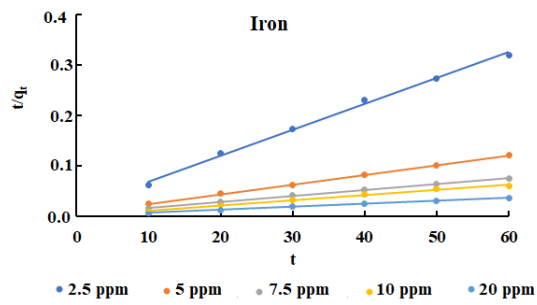
**Table 2: Kinetic parameters of the adsorption of: Fe, Cu, Ni and Zn on CPL**

Metal	C <sub>0</sub> (mg/L)	q <sub>e,exp</sub> (mg/g)	Pseudo-premier ordre			Pseudo-second ordre			Intraparticle diffusion			Elovich		
			ln(q <sub>e</sub> - q <sub>t</sub> ) = ln q <sub>e</sub> - k <sub>1</sub> t			$\frac{t}{q_t} = \frac{1}{k_2 q_e^2} + \frac{1}{q_e} t$			q <sub>t</sub> = k <sub>i</sub> t <sup>2</sup> + I			q <sub>t</sub> = $\frac{1}{\beta} \ln \alpha \beta + \frac{1}{\beta} \ln t$		
			k <sub>1</sub> (min <sup>-1</sup> )	q <sub>e,cal</sub> (mg/g)	R <sup>2</sup>	k <sub>2</sub> (g/mg/min)	q <sub>e,cal</sub> (mg/g)	R <sup>2</sup>	k <sub>i</sub> (mg/g/min)	I	R <sup>2</sup>	β (g/mg)	α (mg/min)	R <sup>2</sup>
Copper	2.5	110.21	0.017	25.96	0.646	0.003	119.05	0.999	4.66	81.70	0.698	0.08	2.46 × 10 <sup>3</sup>	0.761
	5	208.82	0.004	18.12	0.125	0.003	222.22	0.994	2.70	197.27	0.427	0.16	1.09 × 10 <sup>4</sup>	0.351
	7.5	307.43	0.015	23.68	0.207	0.002	322.58	0.998	5.55	275.75	0.735	0.07	1.02 × 10 <sup>9</sup>	0.742
	10	469.84	0.034	92.26	0.493	0.001	500.00	0.997	16.55	345.39	0.872	0.02	4.30 × 10 <sup>4</sup>	0.880
	20	469.84	0.016	57.05	0.311	0.006	454.55	0.993	7.46	396.33	0.231	0.04	2.20 × 10 <sup>9</sup>	0.32
Zinc	2.5	103.42	0.021	4.27	0.419	0.023	103.09	0.999	1.18	94.56	0.528	0.30	1.42 × 10 <sup>12</sup>	0.651
	5	293.67	0.076	286.60	0.726	0.0002	344.83	0.993	23.54	113.95	0.728	0.02	1.44 × 10 <sup>2</sup>	0.662
	7.5	201.74	0.023	39.06	0.251	0.005	192.31	0.991	-0.73	190.58	0.011	-0.28	-	0.039
	10	505.62	0.024	71.93	0.788	0.0007	526.32	0.997	13.25	402.40	0.885	0.03	2.18 × 10 <sup>6</sup>	0.833
	20	352.40	-0.006	67.61	0.205	-0.001	256.41	0.985	-18.16	383.72	0.619	-0.02	-	0.719
Nickel	2.5	55.05	0.004	11.31	0.307	-0.009	44.05	0.986	-0.59	51.90	0.037	-1.54	-	0.007
	5	69.95	-0.030	3.40	0.782	-0.004	48.54	0.994	-4.52	86.59	0.913	-0.09	-	0.865
	7.5	152.52	-0.056	1.27	0.761	-0.002	119.04	0.989	-6.69	176.54	0.673	-0.06	-	0.622
	10	269.49	-0.009	16.46	0.316	-0.002	232.56	0.997	-3.97	272.07	0.322	-0.12	-	0.220
	20	103.21	-0.005	27.26	0.112	-0.004	63.69	0.999	-6.86	113.70	0.544	-0.05	-	0.655
Iron	2.5	187.61	-0.04	50.01	0.850	0.002	196.08	0.997	6.09	139.00	0.914	0.07	4.94 × 10 <sup>4</sup>	0.856
	5	499.50	-0.09	225.61	0.936	0.001	526.32	1.000	18.41	368.32	0.887	0.02	2.73 × 10 <sup>4</sup>	0.942
	7.5	807.15	-0.06	291.20	0.920	0.0003	833.33	0.998	30.80	572.40	0.973	0.01	3.53 × 10 <sup>4</sup>	0.966
	10	938.05	-	-	-	0.001	1000.00	0.993	-1.93	973.44	0.006	-0.09	-	0.033
	20	1668.85	-	-	-	0.012	1666.67	1.000	1.07	1668.20	0.092	0.35	4.42 × 10 <sup>250</sup>	0.103

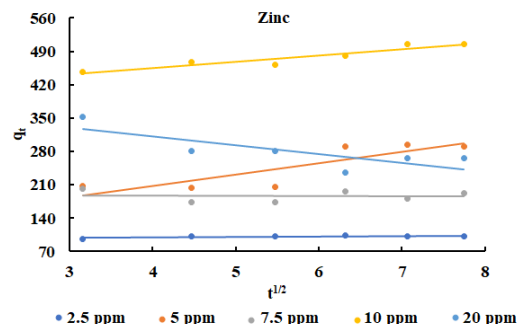
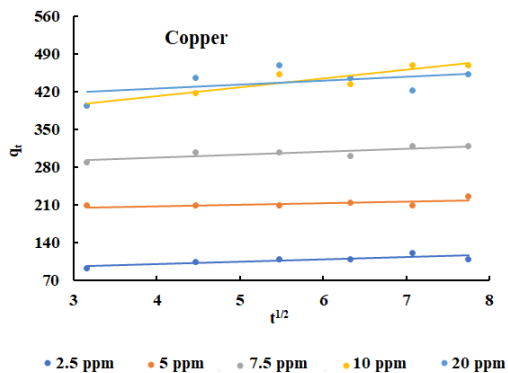
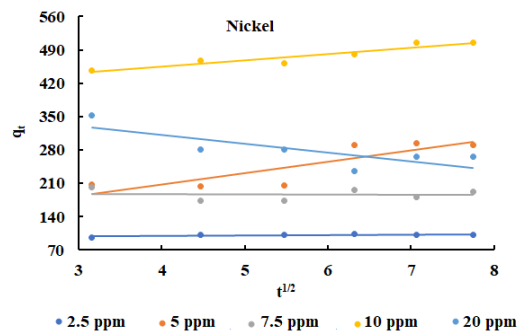
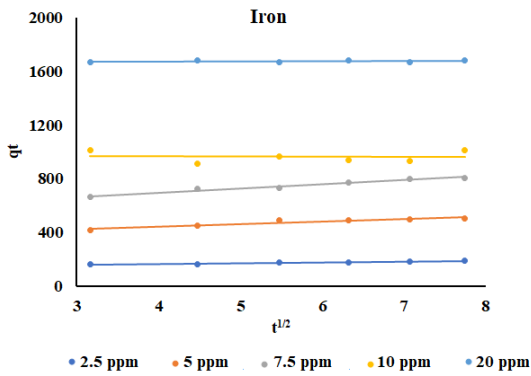


(a)

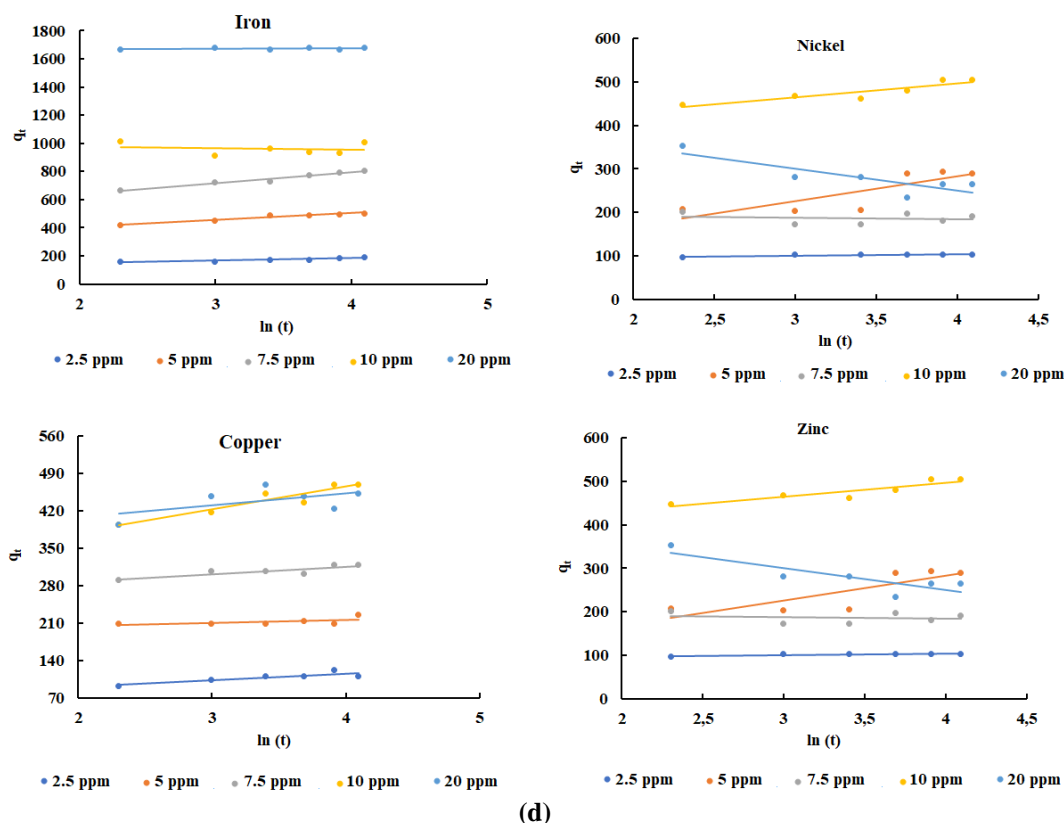




(b)



(c)



**Figure 5 (a-d):** Kinetics models for adsorption of metal on CPL: pseudo-first order (a); pseudo-second order (b); intraparticle (c) and Elovich (d).

#### IV. Conclusion

*Carica papaya* leaves, considered as waste from the papaya sector, constitute an interesting by-product to remove heavy metals in water. From four metals tested, iron is the best removed with an elimination rate reaching 70% under optimal analysis conditions. The other three metals have an elimination rate between 30 and 50%. In this study, the isotherms adsorption was determined for the four metal. The equilibrium of iron and copper are explained by the Temkin model; zinc and nickel equilibriums cannot be explained by the models studied. Adsorption kinetics are described for the four metals and all of them can be explained by the pseudo-second order model. Owing to these results, *Carica papaya* leaves is suitable for cleaning water contaminated by heavy metals.

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