

# Chemical Activation of Charcoal and Application to the Adsorption of Nickel Ions in Aqueous Solution

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## ABSTRACT

The objective of this study was to systematically evaluate the nickel adsorption capacity of carbons that have been chemically activated using phosphoric acid (CAA) and potassium hydroxide (PAC). A series of experimental tests were conducted to optimize the adsorption efficiency of the activated carbons within a controlled environment. The adsorbate consisted of a synthetic nickel solution prepared in distilled water. Experimental results demonstrated that the retention of nickel ions by both CAA and PAC is a reversible adsorption phenomenon. The adsorption efficiency, quantified as percentage adsorption (% Ads), exhibited significant improvement, achieving a maximum adsorption equilibrium after a duration of 60 minutes for both CAA and PAC. Moreover, the pH of the treatment solution exhibited a substantial influence on the adsorption kinetics, with optimal yields occurring at a pH of 6. It was noted that the adsorption efficiency increased proportionally with the mass of the adsorbent employed. Kinetic and isotherm analyses indicated that nickel adsorption adhered to the Langmuir and Freundlich models, suggesting a heterogeneous adsorption

process consistent with multilayer adsorption on the adsorbent surfaces.

**Keywords:** pollution, heavy metals, adsorbent, reversibility and efficiency

## INTRODUCTION

Water pollution, whether accidental or intentional, by certain industrial chemical products constitutes a significant source of environmental degradation and has garnered particular international interest (Mimanne et al., 2014; Rao et al., 2020; Velusamy et al., 2021; Rashid et al., 2021). Contamination of water supply sources by heavy metals poses serious risks to public health and the environment. Heavy metals are toxic even at low concentrations (Ouakouak et al., 2016) and can be distinguished from other pollutants due to their non-biodegradable nature and ability to bioaccumulate in living tissues (An et al., 2001). Several treatment methods have been developed to eliminate these pollutants from wastewater to protect both humans and the environment, including sedimentation, filtration, reverse osmosis, oxidation, photocatalytic degradation, and ion exchange (Mozumder et al., 2010; Piaskowski et al., 2018; Laskar et al., 2018; Atheba et al., 2018; Maiti et al., 2020). However, these conventional treatment

methods have proven to be ineffective for the complete removal of certain pollutants, such as heavy metals. Adsorption processes have demonstrated their efficacy in the elimination of heavy metals (Abollino et al., 2003; Achour et al., 2003; Bouhamed et al., 2012; Larakeb et al., 2015). Activated carbon is currently widely employed as an adsorbent due to its robust capability to retain a wide range of pollutants, a property linked to advancements in its production (Ouakouak et al., 2016; Légbré et al., 2023; Mimann et al., 2014). The objective of this study is to valorize chemically prepared activated carbons, investigate their characteristics, and subsequently assess their performance in the removal of heavy metals. For this purpose, zinc was selected as the target heavy metal due to its presence in numerous industrial effluents. Initially, we will conduct the chemical activation of wood-derived carbons sold in local markets using phosphoric acid and potassium hydroxide. Subsequently, we will contaminate demineralized water and perform zinc removal tests with the two activated carbons for a comparative study.

## **MATERIALS AND METHODS**

### **Materials**

The wood charcoal sold in public markets was utilized as a precursor. After treatment, the charcoal was stored in plastic containers to prevent any external contamination (Bamba et al., 2009; Légbré et al., 2023). The activation of this charcoal was conducted through chemical means. Initially, 30 g of the treated precursor was subjected to impregnation in 300 mL of concentrated phosphoric acid (98 °C) at a 1:1 volumetric ratio of water to acid for a duration of 18 hours. The resulting mixture was then dried in an oven at 105 °C for 24 hours, followed by calcination in an electric furnace at 600 °C for 3 hours. The product was subsequently washed with boiling distilled water and filtered until the pH was within the range of 6 to 7 (Hazourli et al., 2007; Bamba et al., 2009). After this, it was dried in an oven at 110 °C for an additional 24 hours, yielding the activated carbon referred to as CAA.

Conversely, another 30 g of the same previously treated precursor was impregnated with a potassium hydroxide solution at a concentration of 50 g/L for 6 hours before being dried at 110 °C for 24 hours until complete evaporation of the impregnation liquid. Following a similar 24-hour interval, the precursor was placed in a muffle furnace programmed to 400 °C for carbonization for 3 hours (Légbré et al., 2023). After cooling in a desiccator, the charcoal was washed with distilled water under agitation until the pH reached a range between 6.5 and 7. It was then dried in the oven and stored in food-grade bags, resulting in the activated carbon designated as CAP.

### **Methods**

To investigate the influence of adsorbent mass, various quantities (0.2, 0.4, 0.6, 0.8, and 1 g) of activated carbons CAA and CAP were contacted with 50 mL of nickel chloride solution at a concentration of 100 mg/L. The solutions were agitated at 500 revolutions per minute (rpm) for 1 hour and subsequently filtered (Kouadio et al., 2017; Ghedioui, 2020). The residual nickel concentration in the different solutions was determined using Inductively Coupled Plasma Optical Emission Spectroscopy (ICP-OES). To study the adsorption kinetics, 50 mL of nickel chloride solution with an initial concentration of 100 mg/L was mixed with 0.8 g of activated carbon. The mixture was agitated at 500 rpm for 15, 30, 45, 60, and 75 minutes. The residual nickel concentration in the different solutions was also assessed using ICP-OES. For pH optimization, 0.8 g of activated carbon was mixed with 50 mL of a nickel chloride solution having an initial nickel ion concentration of 100 mg/L. Adsorption was conducted at various pH values (2, 3, 4, 5, 6, and 7) by adjusting the pH of the solutions using 0.1 M sodium hydroxide and 0.1 M hydrochloric acid, while maintaining agitation at 500 rpm for 1 hour. The residual nickel concentration in the different solutions was determined by ICP-OES.

## Adsorption Isotherms

**Freundlich Model:** The Freundlich model, a simple and empirical approach, is among the most widely utilized adsorption models. It is applicable in scenarios involving multilayer adsorption, with potential interactions between the adsorbed molecules (Mimanne et al., 2009; Khelifi et al., 2016). The Freundlich equation is expressed as:

$$q_e = K_F \cdot C_e^n$$

The most commonly used form is the logarithmic scale plot of the variations of  $q_e$  as a function of  $C_e$ .

$$\log q_e = \log K_F + n \log C_e$$

The constant  $n$  (dimensionless) gives an indication of the intensity of adsorption.  $K_F$  is a constant which is relative to the adsorption capacity. As  $C_e$  is often expressed in mg/l and  $q_e$  in mg/g, the unit of  $K_F$  is  $mg^{(n-1)} \cdot L^n / g$ .

**Langmuir Model:** The Langmuir model (Senthil et al., 2014; Khelifi et al., 2016) is the second most commonly employed adsorption model. The initial assumptions of this model stipulate that the adsorbent possesses a finite adsorption capacity ( $q_m$ ), that all active sites are homogeneous, that each site can only complex a single solute molecule (monolayer adsorption), and that there are no interactions between the adsorbed molecules. The Langmuir equation can be represented as follows:

$$\frac{q_e}{q_m} = \frac{K_L \cdot C_e}{(1 + k_1 + C_e)}$$

$K_L$ : the Langmuir equilibrium constant, represents the recovery rate and  $q_m$  is the maximum adsorption capacity.

## RESULTS

**Influence of Activated Carbon Mass on Ni<sup>2+</sup> Ion Adsorption:** The figure 1 illustrates the results regarding the influence of the mass of activated carbons, treated with phosphoric acid and potassium hydroxide, on the percentage adsorption of nickel ions (Ni<sup>2+</sup>). The primary objective is to identify the

optimal mass of activated carbon required for the maximum adsorption of nickel ions. The findings reveal that the percentage of nickel ion adsorption increases with the mass of activated carbon. Notably, beyond a mass of 0.8 g, the percentage adsorption (% Ads) remains constant for both types of activated carbons (CAA and CAP). This suggests that there is a saturation point, beyond which additional mass of the adsorbent does not lead to further increases in adsorption efficiency.

**Influence of Contact Time Between Adsorbent and Adsorbate:** The results obtained regarding the effect of contact time between the adsorbent and adsorbate are presented in Figure 2. These findings illustrate that the fixation of Ni<sup>2+</sup> ions onto the adsorbents occurs in two distinct phases. The first phase is rapid, occurring between 0 and 15 minutes, during which the removal rate increases quickly, reaching 55% and 40% for CAA and CAP, respectively. The second phase is slower, starting at 15 minutes, characterized by a gradual adsorption of Ni<sup>2+</sup> until equilibrium is achieved at approximately 45 minutes.

**Influence of pH of the Solution:** The pH is a significant parameter that strongly influences the adsorption of nickel ions onto activated carbon in aqueous solution. The results regarding the influence of pH on the adsorptive solution are presented in figure 3.

**Adsorption Isotherms:** The figures 4, 5, 6, and 7 represent the Freundlich and Langmuir models concerning the adsorption of nickel ions in aqueous solution by the acid-activated carbon (CAA) and the base-activated carbon (CAP). The results indicate that the correlation coefficients for the Freundlich and Langmuir models using acid-activated carbon are higher than those obtained for the models using base-activated carbon. This finding is consistent with previous results, as the adsorption capacity of acid-activated carbon is superior to that of base-activated carbon.

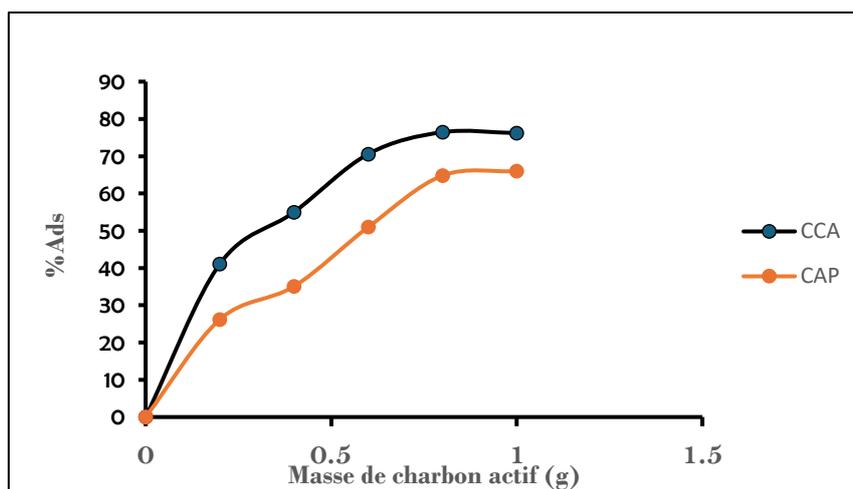


Figure 1: Influence of the mass of carbons activated by phosphoric acid (CAA) and potash (PAC) on the adsorption of nickel ions.

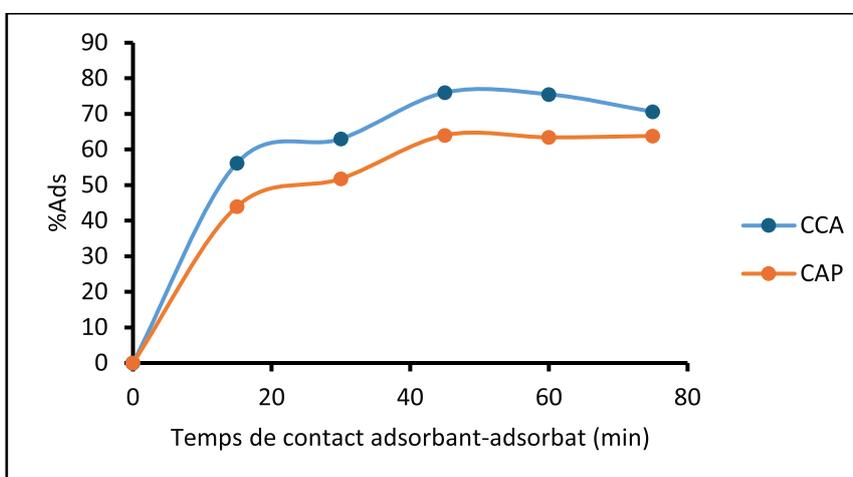


Figure 2: Influence of adsorbent-adsorbate contact time  $[\text{Ni}^{2+}]_0=100 \text{ mg/L}$ ,  $V=50 \text{ ml}$  and  $m=0.8 \text{ g}$

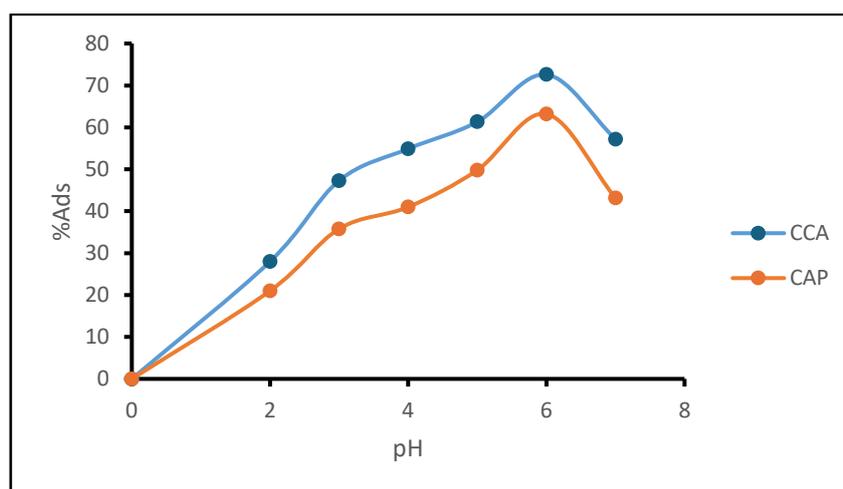


Figure 3: Influence of the pH of the solution  $[\text{Ni}^{2+}]_0=100 \text{ mg/L}$ ,  $V=50 \text{ ml}$  and  $t=45 \text{ min}$ .

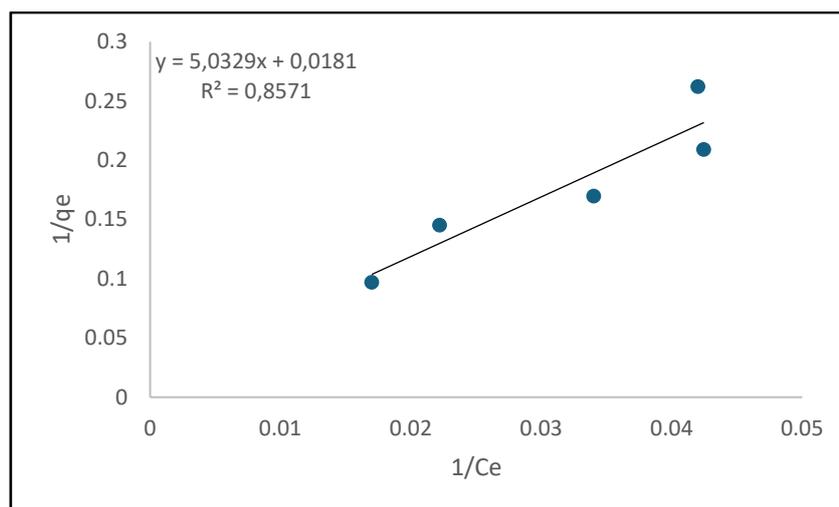


Figure 4: Langmuir model for  $Ni^{2+}$  adsorption on CAA

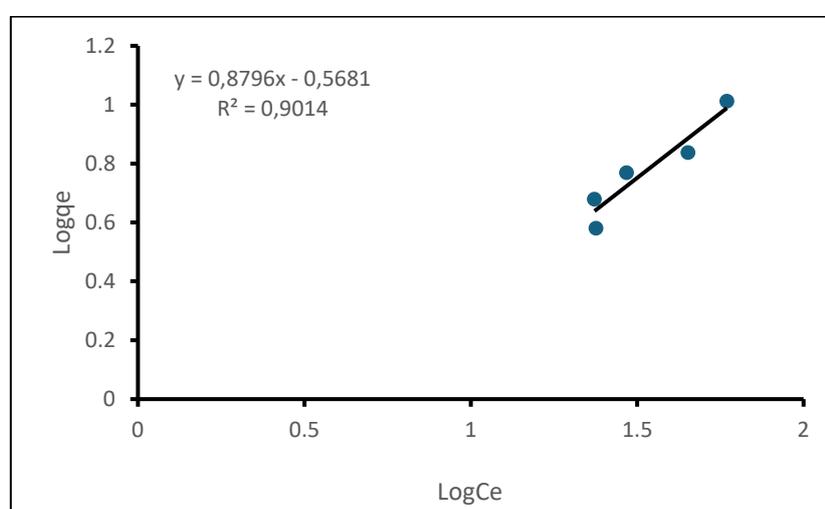


Figure 5: Freundlich model for  $Ni^{2+}$  adsorption on CAA

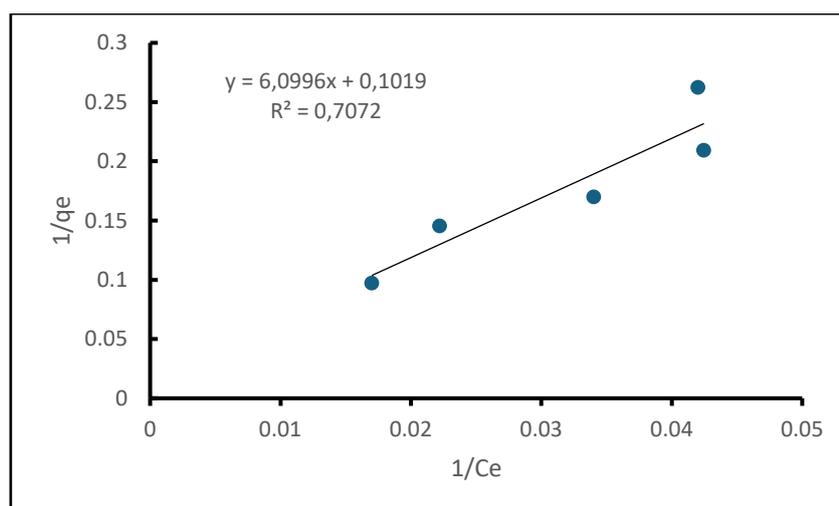


Figure 6: Modèle de Langmuir pour l'adsorption de  $Ni^{2+}$  sur le CAP

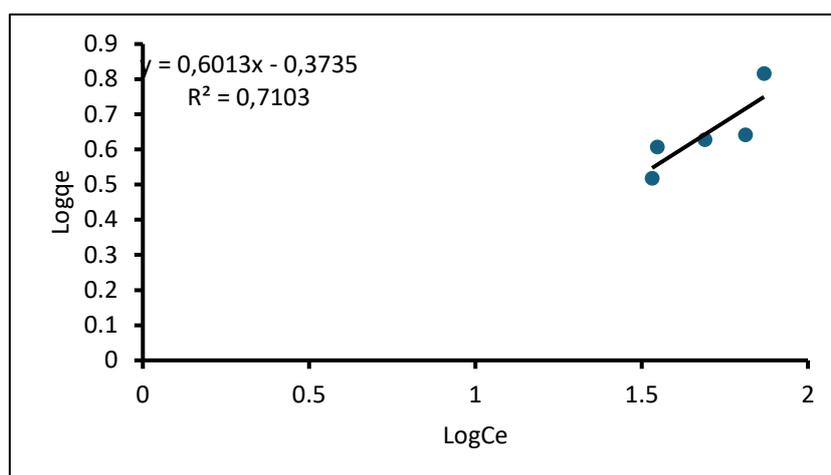


Figure 7: Freundlich model for Ni<sup>2+</sup> adsorption on CAP

Table 1: Table of Freundlich and Langmuir adsorption model constants for phosphoric acid (H<sub>3</sub>PO<sub>4</sub>) and potash (KOH) activated carbons.

Adsorbat	Adsorbant	Isotherme de Langmuir				Isotherme de Freundlich		
		q <sub>max</sub> (mg/g)	K <sub>L</sub> (L/mg)	R <sub>L</sub>	R <sup>2</sup>	K <sub>f</sub>	n	R <sup>2</sup>
Ni <sup>2+</sup>	Charbon actif (CCA)	144,93	0,00123	0,89	0,9041	0,318	1,194	0,9228
	Charbon actif (CAP)	10	0,016	0,38	0,7069	0,4233	1,66	0,7101

## DISCUSSION

The adsorbent dose is one of the most critical parameters influencing the adsorption process, serving as a determinant of adsorption capacity (Diop et al., 2022a). The results indicate that the adsorption rate increases with the mass of activated carbon, a phenomenon that may be attributed to intrinsic differences in the properties of the carbons (Berdji et al., 2023). The increase in carbon mass from 0.2 to 0.8 g correlates with an enhancement in the adsorption rate of Ni<sup>2+</sup> ions for both types of activated carbon. This progression is partly due to the increased exchange surface area, which accompanies a greater number of available adsorption sites (Diop et al., 2022b). Beyond 0.8 g, further increases in the mass of the adsorbent exert minimal impact on nickel ion removal for both carbon types. This behavior may be explained by particle agglomeration, which leads to a reduction in the specific surface area of the adsorbent. Such observations have been reported by several authors (Mimanne et al., 2009; Benamraoui, 2014).

The rapid elimination observed during the initial minutes of adsorption may be attributed to the high availability of active sites on the adsorbent surface at the onset of the process (Shu et al., 2018; Diop et al.,

2022a). These sites decrease over time, leading to a subsequent slowdown in adsorption rates. The slow adsorption phase observed after 45 minutes could be explained by the diffusion of Ni<sup>2+</sup> ions toward adsorption sites until equilibrium is reached, where all sites become occupied and exhibit similar behavior toward nickel ions, or due to the establishment of steady-state conditions (Pova et al., 2015; Xiaoto et al., 2015; Légbré et al., 2023).

Examination of the data indicates that at strongly acidic pH levels, adsorption is negligible. This may be attributed to high concentrations of H<sup>+</sup> ions in solution, which compete with Ni<sup>2+</sup> ions for the available free sites on the presumed negatively charged adsorbent surface. As a result, electrostatic repulsion occurs between the Ni<sup>2+</sup> cations and the positively charged surface (Chouchane et al., 2015). At moderately acidic pH levels, the concentration of protons decreases, while that of Ni<sup>2+</sup> cations remain constant, which accounts for the remarkable increase in the adsorption rate until maximum adsorption is achieved. At pH values greater than 6, the adsorption rate declines as it approaches the precipitation pH of Ni<sup>2+</sup> ions (Olugbenga et al., 2014; Sudha et al., 2015).

The results presented in the aforementioned table demonstrate a higher maximum adsorption capacity for nickel ions using phosphoric acid-activated carbon. Thus, it can be concluded that acid-activated carbon exhibits superior adsorption performance for nickel ions compared to potassium hydroxide-activated carbon. Furthermore, the adsorption intensity is observed to be greater than 1 for both activated carbons, indicating that adsorption is favorable in both cases. This conclusion is supported by the separation factor ( $R_L$ ), with values falling between 0 and 1 for both cases, confirming that adsorption is indeed favorable (Ikuoriel et al., 2005). Additionally, we observe that the correlation coefficients for the Freundlich model are slightly higher than those for the Langmuir model. Consequently, we can assert that the Freundlich model is better suited for describing the adsorption of nickel ions onto activated carbon. In summary, the adsorption of  $Ni^{2+}$  ions onto both phosphoric acid and potassium hydroxide activated carbons occurs on heterogeneous surfaces (Senthil et al., 2010; Thamilarasu et al., 2011).

## CONCLUSION

In this study, commercially available wood-based carbons were utilized as precursors for the removal of nickel. The optimal experimental parameters for nickel adsorption were determined to be an initial pH of 6, a contact time of 60 minutes, an initial concentration of 100 mg/L, and a temperature of 25 °C. The mass of activated carbon is a critical parameter to consider, as our results indicated that the optimal mass for enhanced adsorption is 0.8 g. The pH of a solution and the contact time between the adsorbent and adsorbate play significant roles in the adsorption of heavy metals onto activated carbon. Specifically, the results regarding the effect of pH on the adsorption of nickel ions in aqueous solution demonstrated that the maximum adsorption rate occurs at a pH of 6, with an optimal contact time of 45 minutes. In summary, for effective removal of nickel ions from water,

it is preferable to use phosphoric acid-activated carbon as the adsorbent, as it exhibits a substantially higher maximum adsorption capacity. The Freundlich isotherm is highly recommended due to its superior correlation coefficient compared to the Langmuir model.

## Declaration by Authors

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**Conflict of Interest:** No conflicts of interest declared.

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