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Non-Thermal Plasma Desorption of Copper (II) Ions from a Cu(II)-Clay Composite and Recuperation of the Adsorbent Part B

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Abstract

The regeneration of the adsorbents and recovery of heavy metals are of crucial importance in assessing their potential for commercial application. In this study, Non-thermal plasma (NTP) Glidarc was applied as desorbing agent for Cu(II) desorption from Cu(II)-clay composite. The kinetics was also studied and the comparison with other desorbing agents was performed. Results showed that, NTP Glidarc efficiently removed adsorbed Cu(II). Cu(II) desorption process by NTP occurred following three major steps: a fast initial desorption step followed by a slow desorption step and finally the equilibrium. The increase of the stirring rate and the Cu(II)-clay composite dosage also increases the efficiency of desorption. The equilibrium was reached after 180 min and 99.79% of Cu(II) was recovered. A comparison with other desorbing agents followed the order: plasma > HCl > $CaCl_2$ > NaCl. Six adsorption-desorption cycles did not significantly affect the adsorption capacity of the clay. Desorption with NTP and HCl fitted well the first order, Elovich, parabolic diffusion and modified Freundlich kinetic models. The time of half reaction was 45 min for NTP and 61 min for HCl. NTP can thus be considered as an efficient heavy metal desorption strategy from adsorbents.

Keywords

Desorption, Non-Thermal Plasma, Clay, Copper

1. Introduction

The fast development and expansion of industries have led to the presence of heavy metals in the vital environment and resources which have become a major threat for plant, animal and human beings due to their bioaccumulating tendencies and toxicity (Camp, 1964). Adsorption is one of the best methods used to control them (Babel & Kurniawan, 2003; Dias et al., 2007). Many studies reported the high efficiency of activated carbons, biomass wastes, clay minerals, magnetic polymers and others for the removal of heavy metals from wastewaters by adsorption (Babel & Kurniawan, 2003; Dias et al., 2007; Fu & Wang, 2011; Demirbas, 2008, Bhattacharyya & Gupta, 2008; Gupta & Rastogi, 2008). This technique of waste control translocates the heavy metals from aqueous phase where they cannot be easily controlled to a solid phase where they can be easily controlled or recovered for further uses. However, saturation of adsorbent remains the biggest problem (Fouodjouo et al., 2017) and needs to be solved. Indeed, regeneration of spent adsorbents and recovery of heavy metals for further uses are strongly important to keep low processing cost for heavy metals pollution control (restoration of the adsorbent) and give the possibility of reuse of the adsorbate (heavy metal) in the industrial production process. The heavy metal adsorbed onto an adsorbent can be desorbed by a suitable desorbing agent or eluant and reused in multiple sorption-desorption cycles. Several techniques such as proton exchange (HCl, HNO_3, NaOH), Chelation (EDTA, NTA), cationic exchange (NaCl, $CaCl_2$), bleaching with deionized water, microorganisms and thermal treatment (Microwave, thermobath) in batch or continuous flow systems (Bayuo et al., 2020; Kwon & Jeon, 2012; Wankasi et al., 2005; Lo et al., 2003; Motaghian & Hosseinpur, 2013) have been used with relative efficacies and often onerous or pollutant. The choice of desorbing agent depends on the nature of adsorbent and the metal loaded. The researches of effective desorbing agents that desorb without affecting the nature of the adsorbent and adsorbate remain a challenge of this century. Previous studies report the uses of non-thermal plasma for desorption of methylene blue, isopropyl alcohol, toluene on adsorbents with high efficiencies (Ma et al., 2019; Vierck & Leclerc, 2017; Shiau et al., 2017; Sivachandiran et al., 2014; Sivachandiran et al., 2013a, Sivachandiran et al., 2013b). Besides, NTP has shown its high ability for regeneration of solid catalysts by desorption in gaseous phases (Pinard & Batiot-Dupeyra, 2024; Zeng et al., 2017; Srour et al., 2021, Okubo et al., 2002). NTP in humid air is known to be an environmentally friendly source of excited and highly reactive radicals (NO•, •OH) created in the electrical discharge without using any additional reagents (Benstaali et al., 1999; Czernichowski et al., 1996; Benstaali et al., 2002). The contact of the plasma plume with aqueous media enriched the suspension with acidifying and oxidizing properties evidenced by previous works (Brisset & Hnatiuc, 2012). The oxidizing property of aqueous suspension after interaction with NTP in humid air is related to the presence of H_2O_2 in the suspension while the acidifying property is related to the presence of nitric acid (HNO_3) and peroxonitrous acid (ONOOH) (Brisset & Hnatiuc, 2012). Non-thermal plasma with humid air as feeding gas is particularly cheap in term of energy and processing and the dispositive is easy to build. The combination of the chemical properties, the cleanliness and the low cost makes NTP a suitable desorbing technique for adsorbent regeneration and heavy metals recovery by desorption.

This work reports a feasible regeneration of adsorbent (Cameroonian clay) and recovering of heavy metal (Copper (II) ions) in aqueous suspension assisted by Gliding arc plasma using Humid air as feeding gas.

2. Materials and Methods

2.1. Desorption Experiments

The clay material used for adsorption experiments performed in the previous study is essentially kaolinite and was collected from Konhontsa'a, a small town in Bamboutos Division located in the West Cameroon Region (5˚41"N and 10˚13"E) (Fouodjouo et al., 2017). The adsorption experiments were carried out in 250 mL Erlenmeyer flasks by mixing together 0.1 g of clay with 50 mL of aqueous solution of Cu^{2+} of concentration 3 $g \cdot L^{-1}$ (Fouodjouo et al., 2017). The contents in the flasks were agitated by placing them in a constant temperature water bath thermostat for 3 minutes (equilibrium time) (Fouodjouo et al., 2017). The mixture was then centrifuged (4000 rpm) and residual Cu^{2+} in the supernatant liquid was determined by atomic absorption spectroscopy (Fouodjouo et al., 2017). The Cu(II)-clay composite samples used are those corresponding to maximum adsorption capacity at equilibrium found in part A of this strudy by Fouodjouo et al. (2017).

Desorption using NTP or desorbing was carried out in batch system and the temperature was kept at 298 K using a cooling bath system. 1 g of natural dried Cu(II)-clay composite (KON-00, q_0 = 195 $mg \cdot g^{-1}$) (Fouodjouo et al., 2017) was dispersed in 400 mL of deionised water and the suspension introduced into the Glidarc plasma batch reactor presented in **Figure 1**. Humidified air was used as flowing gas at a flow rate of 800 $L \cdot h^{-1}$. The homogenisation of the suspension in the reactor was assured by the airflow and a magnetic stirrer. The Glidarc device used (**Figure 1**) operation is described by Fouodjouo et al. (2017) and the temperature and electron density, and the distribution of active species in the plasma by Wu et al. (2015).

The effect of exposure time, initial amount of Cu(II)-clay composite and stirring rate on the efficiency of the desorption process were studied. Furthermore, for a comparative study with other desorbing agents, 1 g of Cu(II)-clay composite was placed into flasks of 200 mL containing 50 mL of analytical grade aqueous solutions of $CaCl_2$, HCl or NaCl respectively. The selected concentrations of desorbing agents were 0.1 $mol \cdot L^{-1}$, 0.01 $mol \cdot L^{-1}$ and 0.001 $mol \cdot L^{-1}$. The temperature was maintained constant by using a thermostated bath. Aliquots drawn after each of time intervals chosen, were centrifuged at 3500 rpm for 5 min and the concentration of Cu(II) ions in the supernatant was determined using atomic adsorption spectroscopy (Buck scientific 200A; Atomic Adsorption/Emission spectrophotometer). The clay and the Cu(II)-clay composite were characterized in the previous works (Fouodjouo et al., 2017).

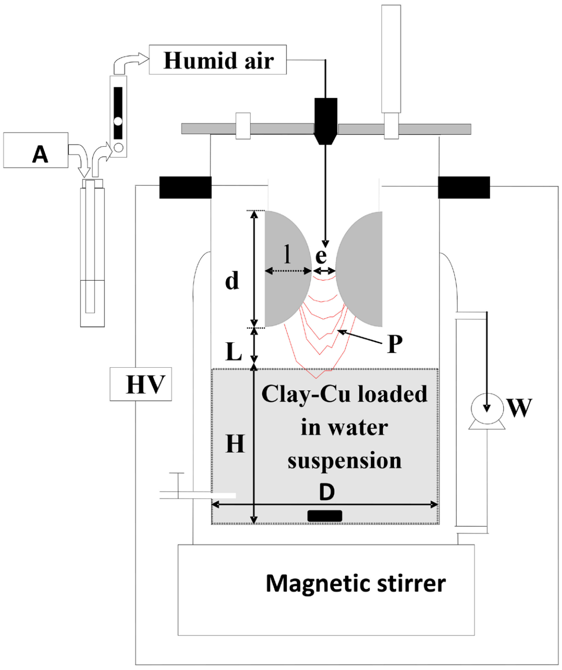

Figure 1. Experimental setup of Glidarc plasma (A = air entrance; E = inter-electrode gap of 3 mm; I = electrode ray of 2 cm; d = electrode length of 7 cm; L = distance between electrodes and target surface of 1.5 cm; D = reactor diameter of 8 cm; H = height of solution in the reactor 10 cm; W = water pump; HV = high voltage).

2.2. Calculation

The amount of Cu(II) desorbed per unit mass of clay and the percentage of Cu^{2+} recovering as function of time were estimated using Equations (1), (2) and (3) presented below (Wankasi et al., 2005; Wambu at al., 2009):

$$q_{Des} = C_t \left(\frac{V}{m} \right) \quad (1)$$

$$q_t = q_0 - q_{Des} \quad (2)$$

$$\% Des = \frac{C_t}{10 m q_e} V \quad (3)$$

where q_{Des} and q_t in $mg \cdot g^{-1}$, are respectively the amount of Cu(II) desorbed per unit mass of copper-loaded clay and the amount of remaining Cu(II) onto the Cu(II)-clay composite at time t (min), C_t ($mg \cdot L^{-1}$) the concentration of Cu(II) in desorption solution at time t and q_0 ($mg \cdot g^{-1}$) the initial solid phase Cu(II) concentration.

2.3. Kinetic Modelling

Four kinetic models (Table 1) were applied to desorption data and their respective parameters were determined.

Table 1. Different kinetic Models used in this study.

Kinetics model	Equation	Parameters	References
First order	$\ln q_t = b - k_1 t$	k_1 is the apparent desorption rate coefficient (min^{-1}), b a constant	Elkhatib et al., 2007a; Wambu et al., 2009
Elovich	$q_t = \frac{1}{\alpha}\ln\alpha + \frac{1}{\alpha}\ln t$	a is a constant, α a constant related to initial rate of reaction	(Tsé & Lo, 2002; Chien & Clayton, 1980
Parabolic diffusion	$q_t = C + k_d t^{\frac{1}{2}}$	C is a constant and k_d the apparent diffusion rate constant.	Elkhatib et al., 2007a
Modified Freundlich	$\ln q_t = \ln k_D C_i + \frac{1}{m}\ln t$	k_D is the desorption rate coefficient and $1/m$ a constant	Reyhanitabar & Karimian, 2008

where q_t is the Cu(II)-clay composite concentration (mg·g^{-1}) at any time t(min) and C_i the initial Cu(II) ions concentration loaded on Cu(II)-clay composite.

3. Results and Discussion

3.1. Influence of Plasma Exposure Time and pH

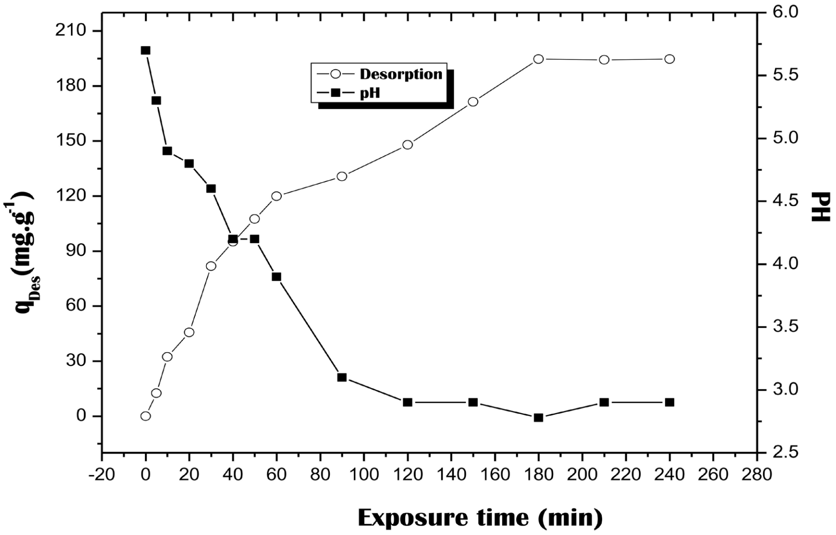

Figure 2. Influence of exposure time and pH on desorption of Cu(II) ions onto clay Cu(II)-clay composite (q_0 = 195 mg·g^{-1}, T = 298 K, m = 1 g, 500 rpm).

Figure 2 displays the effect of plasma exposure time on the Cu(II) desorption from a Cameroonian clay. It appears that the amount of Cu(II) in the composite decreased with the increase of the exposure time, followed by the formation of a plateau at 180 min corresponding to a 99.79% Cu(II) removal. Desorption with NTP can be explained by the collision of fast active species generated with copper ions (Shiau et al., 2017; Ghosh et al., 2015; Johnson, 1987). The composite Cu(II) concentration also decreased with the decrease in solution pH of extraction solution (Wankasi at al., 2005) caused by the acidifying effect of Glidarc plasma in humid air (Brisset & Hnatiuc, 2012). Indeed, the humid air NTP is a source of H$^+$

ions (Njoyim-Tamunganga et al., 2011; Brisset & Hnatiuc, 2012), which can easily exchange with Cu^{2+} ions fixed on the adsorption sites of the adsorbent due to their small size and high mobility. The prevailing reaction at the surface of metal-loaded adsorbent in acidic medium and responsible for Cu(II) desorption can be described as follows:

$$\left(\mathrm{Ads}^{2-}\right)\left(\mathrm{M}^{2+}\right)+2\mathrm{H}^{+}\rightarrow\left(\mathrm{Ads}^{2-}\right)\left(2\mathrm{H}^{+}\right)+\mathrm{M}^{2+} \quad (3)$$

where $(\mathrm{Ads}^{2-})(\mathrm{M}^{2+})$ is the metal-loaded adsorbent and, M^{2+} the metal.

This means that loaded copper onto clay could be in form of pH-susceptible speciation such as hydrolysis products. The oxidative species such as OH• and H_2O_2 might also participate to the breaking of the bonds between Cu(II) ions and the adsorbent. The equilibrium was reached after 180 min and 99.79% of Cu(II) was recovered from the metal-laden clay.

3.2. Influence of Stirring Rate

The effect of the stirring rate of the desorption medium on Cu(II) desorption is depicted in Figure 3. The results obtained suggests that, Cu(II) desorption efficiency increased with increase of stirring rate. This signifies that, stirring could speed up the hydrogen ions or the oxidative species diffusion and thus accelerated the displacement of Cu(II) ions from the solid phase. The increasing of the stirring rate reduced the time necessary for complete desorption. At 1250 rpm, 87.97% of copper was desorbed after only 30 min.

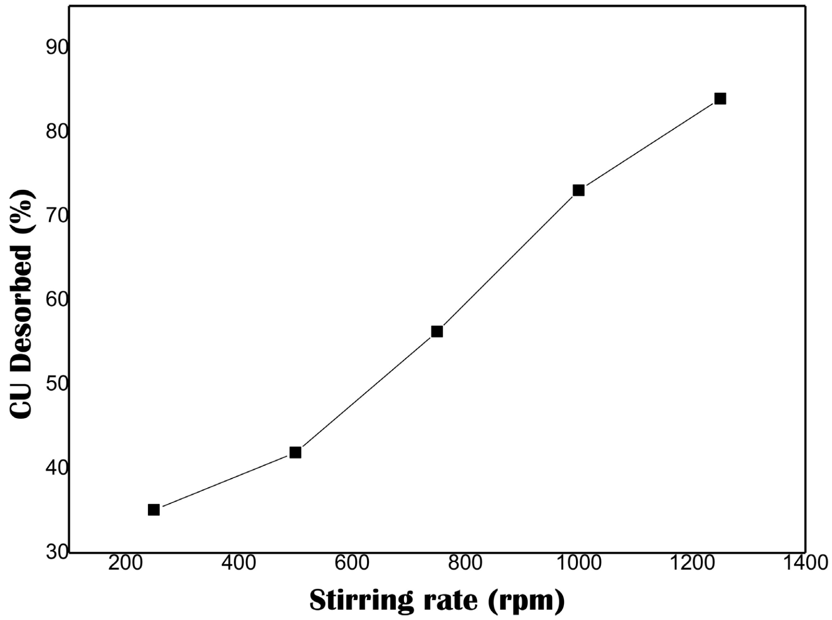

Figure 3. Influence of stirring rate on desorption of Cu(II) ions onto clay Cu(II)-clay composite (desorption time 30 min, $T = 298$ K, $m = 1$ g, $q_0 = 195$ mg·g^{-1}, 500 rpm).

3.3. Influence of the Dosage of Cu(II)-Clay Composite

Figure 4 Presents the effect of Cu(II)-clay composite dosage on the Cu(II) desorption. The efficiency of desorption increased with the increase of the composite dosage. The maximum was reached for adsorbent dosage of 1.5 g. Above this value, equilibrium of desorption was established despite the increase of the adsorbent dosage. The porosity of Konhontsa'a clay coupled with big S_{BET} is helpful in expanding the discharge region because the streamers could be generated on solid surface with such system (adsorbent with NTP). Further, plasma performance could be enhanced. Kim et al. (2008) report that the metal supported material results in a larger discharge region compared with the unsupported one. In other words, surface properties could indeed affect discharge behavior. Therefore, collision chance Cu^{2+} ions with energetic electrons and excited species could be increased, resulting in higher desorption concentration as found for isopropyl alcohol by Shiau et al. (2017).

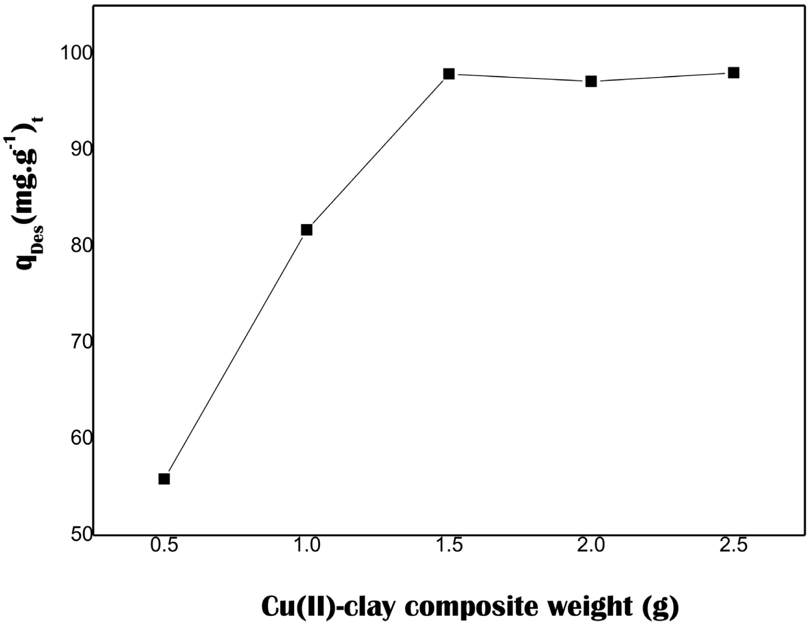

Figure 4. Effect of Cu(II)-clay composite dosage (desorption time 30 min, $T = 298$ K, $q_0 = 195$ mg·g^{-1}).

3.4. Adsorption-Desorption Cycles with Plasma as Desorbing Agent

Reusability of the clay has been checked via six adsorption-desorption cycles with NTP plasma as desorbing agent. **Figure 5** shows that after six cycles the adsorption capacity of the cycles decreased only of 38.97%. The adsorption capacity dropped from 195 mg·g^{-1} to 119.23 mg·g^{-1}. The use of NTP as desorbing agent did not noticeably change the adsorption capacity of the adsorbent and thus, the clay could be repeatedly used in Cu(II) ions adsorption without significant loss of initial adsorption efficiency. This is due to the fact that NTP at each cycle increase the oxygen content on the adsorbent surface (Zeng et al., 2017). Indeed, several previous works reported that the plasma does not significantly affect the adsorption capacity of the clay (Pinard & Batiot-Dupeyra, 2024; Srour et al., 2021). NTP plasma is suitable to keep low the cost of Adsorption-desorption processing and therefore the overall cost of heavy metal control in water using Adsorption technique.

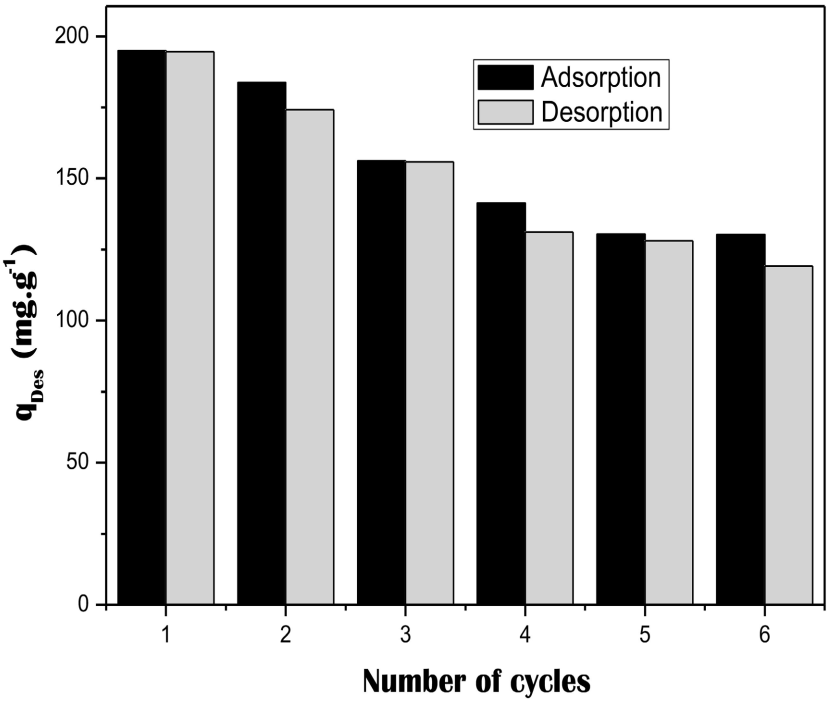

Figure 5. Adsorption-desorption cycles (C_i = 3000 mg·L^{-1}, T = 298 K, 500 rpm, m_{Ads} = 0.1 g, V_{Ads} = 50 mL, pH_{Ads} = 5.7).

3.5. Kinetics of Desorption

Kinetics study of Cu(II) desorption from Copper loaded clay using NTP as desorbing agent was important to understand the process. As shown in Figure 2 in section 3.1, the desorption rate was rapid during the first 60 minutes, then, it slowed down until the desorption process approached completion. The desorption attained equilibrium in 180 min at a stirring rate of 500 rpm. The kinetics of Cu(II) desorption by NTP and HCl as desorbing agents were evaluated using the first order, Elovich, modified Freundlich and parabolic diffusion equation models. These multiple models were applied to point out the efficiency of NTP compared to others desorbing efficient agent such as HCl. The kinetic parameters obtained were confined in Table 2.

First order kinetics

Figure 6(a) shows the plot of lnq_t versus t at 298 K. It appeared that the experimental data fitted linear with first order model. The first order equation assumes that the rate of Cu desorption is proportional to either the number of occupied sites on the surface or the concentration of Cu in the occupied sites. The first order equation constants (k_1 and b) and the correlation factor R^2 were determined and presented in Table 2. From those constants it can be seen that NTP was more efficient and faster than HCl for Cu(II). Furthermore, the half time of desorption $t_{1/2}$ was found to be 45 min for NTP and 61 min for HCl.

Elovich kinetics model

The empirical Elovich equation (Chien & Clayton, 1980; Kuo & Lotse, 1973) was also used to describe the Cu desorption rate. **Figure 6(b)** depicts the plot of qt versus logarithm of exposure time at 298 K. It showed a linear relationship exists between q_t and lnt for desorption with NTP as desorbing agent. **Table 2** recapitulated all the elovich parameters for Cu(II) desorption using NTP and HCl as desorbing agent. In fact, a decrease in α value, evidences a reduction in the desorption reaction rate (Motaghian & Hosseinpur, 2013). The α value of NTP as desorbing agent was higher than that of HCl, showing the high capacity of this environmentally friendly technique (NTP) to be the clean and efficient desorbing agent for heavy metals.

Parabolic diffusion

Parabolic Diffusion Equation was also used to describe the kinetic data of Cu(II) release with NTP or HCl as desorbing agent. The plot of q_t against $t^{1/2}$ at 298 K (**Figure 6(c)**) was linear, showing that experimental data fitted well this kinetic model. Diffusion law parameters determined from the slope and intercept of the linear plots are shown in **Table 2**. The absolute value of parabolic diffusion rate constant of desorption with NTP is once higher that of HCl.

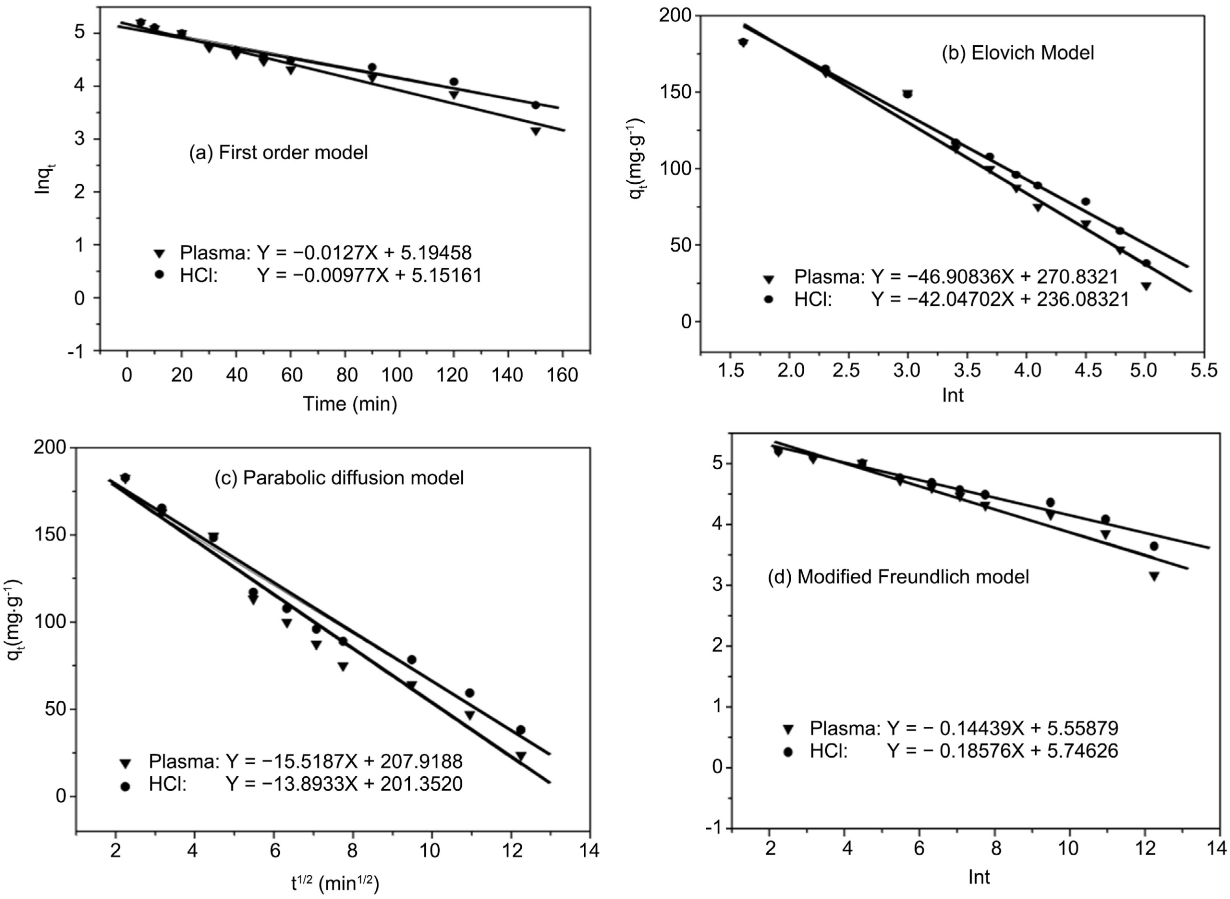

Figure 6. Kinetics of desorption of Cu(II) from Cu(II)-clay composite using Glidarc plasma and HCl 0.1M (500 rpm, q_0 = 195 mg·g^{-1}).

Table 2. kinetic parameters of Cu(II) desorption from copper-loaded clay (HCl 0.1M, 500 rpm, q_0 = 195 mg·g^{-1}).

Kinetics models	Constants	Desorbing agents	
		Plasma	HCl
First order	k_1 (min^{-1})	0.0127	0.0098
	b	5.1945	5.1516
	R^2	0.966	0.963
Elovich	a	−16095.32	−20884.83
	α	−0.0200	−0.0237
	R^2	0.966	0.971
Parabolic diffusion	k_d	−15.518	−13.893
	C	207.92	204.35
	R^2	0.962	0.964
Modified Freundlich	k_D	0.1145	0.0888
	$1/m$	−0.1857	−0.1444
	R^2	0.944	0.963

Modified Freundlich kinetics

The Modified Freundlich Equation was also used to describe the Cu desorption data. The plot of lnq_t versus lnt at 298K (**Figure 6(d)**) gave a straight line. This kinetics models as the three others models studied above fitted well experimental data and can be used for the description of the mechanism of desorption of Cu(II) from metal loaded clay. The modified Freundlich equation parameters k_D and $1/m$, calculated from the linear plots are listed in **Table 2**. From **Table 2**, it can be seen that the k_D of NTP is highest in comparison to that of HCl.

All the four kinetics models studied described well the desorption of Cu(II) from metal loaded clay (R^2 close to 1). Similar observation was done by Elkhatib et al. (Elkhatib et al., 2007b). However, for desorption kinetics of Cu(II) with NTP, experimental data correlated well with the different models studied in the following order: First order > Elovich > Parabolic diffusion > modified Freundlich.

3.6. Comparison of Plasma Desorption with Other Desorbing Agents

The histogram of **Figure 7** depicts the performances of some desorption medium (HCl, CaCl$_2$, NaCl) of various concentration compared to the plasma process. For comparison, desorption was also performed in deionised water. Plasma (99.79%) and HCl (97.63% at 0.1 M) showed the best performances compared to CaCl$_2$ (max value), NaCl (max value) and deionised water (max value). Although HCl had effectiveness close to plasma efficiency, NTP is the best desorbing agent because of its environmental friendliness, its low cost and ease to set up. The most significant difference between NTP desorption process and conventional desorption is the existence of abundant short-lived active species (i.e. electrons, excited species, radicals, and positive/negative ions) (Shiau et al., 2017; Benstaali et al., 2002, Czernichowski et al., 1996). Thus, Cu(II) ions adsorbed can be effectively desorbed and even converted during NTP process. The desorption efficiency followed the order: plasma > HCl > $CaCl_2$ > NaCl > deionised water. From **Figure 5**, it was also found that for chemical desorbing agents, the efficiency of desorption increased with the increase in concentration of the desorbing agent. Earlier, Gupta & Rastogi (2008) and Kwon & Jeon (2012) found the same observation with Cr (VI) and Indium ions respectively.

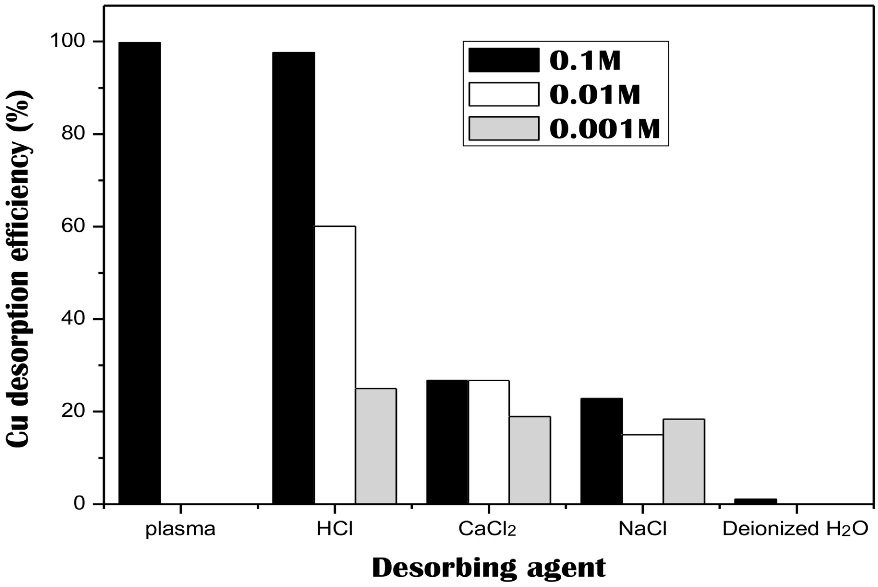

Figure 7. Comparative study of Cu(II) ions plasma desorption efficiency with various desorbing agents at different concentrations (q_0 = 195 mg·g^{-1}, T = 298 K, 500 rpm, t = 240 min).

4. Conclusion

In this study, Glidarc plasma was used as desorbing agent for Cu(II) desorption from a Cameroonian clay in aqueous suspension. Results reported that, NTP is a suitable and clean agent for heavy metal desorption. After six adsorption-desorption cycles by NTP, the adsorption capacity of the clay was not significantly affected. Desorption process of Cu(II) by NTP can be divided into three phases: a rapid desorption phase, slow desorption stage and the equilibrium. Almost all the Cu(II) was desorbed after 180 min of exposure to the discharge. NTP desorption was more efficient than acid or cationic exchange. The stirring rate or the initial mass of metal-loaded adsorbent also increased the efficiency of desorption. Cu(II) desorption by NTP or by HCl fitted all four kinetics models studied. The time of half desorption reaction was found to be 45 min for NTP and 61 min for HCl. Desorption by NTP was faster than desorption with HCl.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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