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Use of x-ray fluorescence for determination of trace metallics elements in water by adsorption on activated carbon

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Abstract

The present work aims to determine heavy metals in textile industry wastewater by using activated carbon and X-ray fluorescence spectrometry. In this work, wastewater from a textile industry located in Bouake /Côte d'Ivoire was studied. The results show that using activated carbon as adsorbent, trace metallic elements can be determined using X-ray spectrometry. The results obtaining are the difference between own element trace metallic containing in poor activate carbon and rich one. From this study, it has been established that contain in trace metallic elements of water from the dam (site P1) is lower than those in water from sites P2 and P3. In addition, waters from sites P2 and P3 present cadmium, nickel, copper, and chromium values above the FAO limit values. On the other hand, waters from the studied sites indicate manganese and zinc values below the FAO limit values. Furthermore, lead and mercury are absent in irrigation water. These different values of trace metallic elements are probably due to the activity of the textile industry, but also to the important use of fertilizers. This study shows that the determination of metallic trace elements by adsorption on activated carbon can be a useful and cheaper solution for determination of metallic trace elements in wastewater. But an optimization of the method is necessary to be definitively useful.

1. Introduction

Food contamination by various pollutants is a major societal concern [1, 2]. This phenomenon has increased since development of industries whose processes are precursors of the pollution [3, 4].

The textile industry is one of the most polluting industries due to the discharge of huge quantities of wastewater loaded with organic and inorganic matter [5, 6]. Indeed, wastewater from processes is generally rich in colorants and may contain trace metallic elements [7, 8]. From a health point of view, metallic trace elements can be responsible for various diseases such as kidney damage, rickets, ulcers, anemia, physiological disorders, and neurological disorders [9–11].

In Bouake, a survey revealed the direct or indirect use of wastewater from the textile industry by market vegetables producers for watering their agricultural plots. Regarding the potentially polluting nature of this irrigation water, the risk of contamination of agricultural soils and subsequently of crops with trace metal elements is real. The evaluation of this risk requires the determination of the contents of metallic trace elements in the irrigation water. Previous studies have showed that the main techniques for the determination of trace metals elements in liquid effluents are atomic absorption spectrometry and inductively coupled atomic emission spectrometry [12, 13]. However, these methods are destructive for samples, require complex preparations and the use of several reagents. In addition, determination of TME (trace metallic elements) by these methods is singular and the process requires several calibrations

of the equipment [14, 15]. Regarding these technical constraints, X-ray fluorescence, an emerging technique in the field of chemical analysis, appears as an alternative for determination of trace metallic elements thanks to its simplicity and its efficiency in determining several TME in one line. Several studies have focused on the use of X-ray fluorescence for determination of metallic trace elements with convincing results. However, these studies, based on the adsorption of metals on commercial materials such as silica gel or gelatin before their determination by X-ray fluorescence, make these analyses expensive [16, 17]. The search for easily accessible and less expensive adsorbent materials seems to be the most important factor in ensuring the routine use of this new analytical technique for the determination of TME in liquid samples instead of using silica gel and other more expensive adsorbents. The present study is a contribution to ensuring the routine use of X-ray spectrometry in determination of metallic trace elements in liquid substances such as wastewater.

2. Material and Methods

2.1 Material

2.1.1 Presentation of the study area and description of sampling sites

The study area is located near a textile industry in the city of Bouake. **Figure 1** below shows the study area and the sites listed under P1, P2 and P3.

Figure 1 : Sampling sites

Site P1 includes a dam that serves as a water reserve for the textile industry. It receives rainwater and is located upstream of the mill's effluent spillway. This water was therefore used as a reference for the study aera.

Sites P2 and P3, downstream from the mill effluent discharge point, include the agricultural plots that are irrigated with water from sumps and from the watercourse receiving untreated mill effluent. The latter are types of water reservoirs on the edge of the watercourses. The water from these two water sources are the targets of this study.

The samples were coded as a function of the site. Thus,

- P1 stands for the waters samples from the dam located upstream of the textile factory.

- P2 and P3 denote water samples from the textile mill and traditional wells from sites P2 and P3 respectively.

2.1.2 Sampling equipment

The sampling equipment from the different matrices consists of:

- a GPS navigator (Global Positioning System) type GARMIN for the location of the sampling points,
- a cooler for the conservation of samples during transportation to the lab,
- Polyethylene bottles with a capacity of 1 liter, free of contamination.

2.1.3 Analytical equipment

The activated carbon used for the adsorption of TME was prepared from coconut shell carbon. It was chemically activated with 3M potash. Its iodine number is 1050 (cc/100 g) with a methylene blue number of 13. The carbon hardness is 98% and has a specific surface area of 147.2 m^2g^{-1}. The granulometry of the carbon used was less than 0.25 mm.

The X-ray fluorescence spectrophotometer used is SPECTRO XEPOS type **(Figure 2)**. This instrument is equipped with a high sensitivity of detection limits that allows to perform multi-element analysis.

Figure 2 : The X-ray fluorescence spectrophotometer SPECTRO XEPOS

During the analysis, the Gas Flow was between 79 and 82 ml.min-1. The voltage of the current varied between 39 and 40 kV, and the current flow was 0.88 mA. The analysis time was 16 min.

2.2 Methods

2.2.1 Analytical equipment

Water samples were collected in February 2018 during the dry season to avoid dilution effects by rainfall. The sampling was done by direct immersion of the bottle previously rinsed three times with the same water. The water samples were taken at a depth of about 30 cm below the water surface. At each

site 10 water samples were taken, stored in a dark place and transported at a temperature of 4°C in a cooler to the laboratory for analysis.

2.2.2 Determination of trace metallic elements

Referring to the use of the X-ray fluorescence method for the determination of metallic trace elements in water using chelating agents or adsorbents such as silica gel, an inexpensive adsorbent alternative as activated carbon was opted for. The principle of the method consists in adsorbing metals from the water sample using an activated carbon followed by X-ray fluorescence analysis of the carbon that has adsorbed the metals. Thus, 10 g of activated carbon was introduced into 100 mL of water samples previously acidified to pH 4 with hydrochloric acid. The mixtures were stirred for 24 hours and the water was slowly evaporated on a sand bath at 50°C.

The carbon was dried in ambient air (around 30°C), ground at 200 µm, then 4 g of carbon powder was mixed with 1 g of FLUXANA CEREOX binder and a pellet was pressed using a 10 tons hydraulic press of the SPECAC ATALS type.

At the same time, a blank test was carried out under the same conditions with acidified distilled water.

The quantification of the metallic trace elements contained in the water samples was made using the following equation: $\boldsymbol{\Delta TME = C_r - C_0}$

- ΔMTE is the amount of TME contained in the water sample.
- C_r is TME containing in the carbon used for water treatment.
- C_0 is TME containing in the carbon from the blank experiment.

2.2.3 Statistical analysis

The statistical analysis of the data obtained was carried out using the open-source software R. The arithmetic means and standard deviations were established and the differences between the groups were calculated using the analysis of variance (ANOVA). When the ANOVA revealed significant differences ($p < 0.05$) between the groups, the Student-Neuwmann-Keuls test was used to determine the specific differences between the means.

3. Results and Discussion

Addition of carbon to the water samples allowed to adsorb / absorb metallic trace elements contained in wastewater. After evaporation of residual liquid, we obtained specimens suitable for XRF analysis of solutions. The metals identification was done based on the built-in data from the apparatus. But for this study a focus was made on the trace metallic elements generally found in textile effluents and those toxic for human beings such as cadmium and mercury.

3.1 Contents of metallic trace elements in carbon

Carbon is generally from biomass and then content already metals such of trace metallic elements. **Table 1** shows the levels of trace metallic elements in initial (P0) and used carbon at the end of the water treatment process.

From **Table 1** we can notice that before using carbon (P0) for water treatment it initially contains 2.15 $mg.kg^{-1}$ of cadmium, 41.4 $mg.kg^{-1}$ of copper, 11.367 $mg.kg^{-1}$ of manganese, 7.2 $mg.kg^{-1}$ of nickel, 7.833 $mg.kg^{-1}$ of zinc and no chromium. The presence of cadmium in initial (P0) carbon, can be due to a contamination of the raw material used to produce carbon, or to a contamination that occurred during its preparation. In the literature, few studies have revealed a natural presence of cadmium in coconuts. Our results are close to those of Begum and Krishna, 2010 [18] obtained in coconut trees in India.

According to these authors, this concentration of cadmium is due to high pollution from anthropogenic activities, resulting from rapid urbanization.

Table 1. Heavy metal concentrations in carbon ($mg.kg^{-1}$)

	Cd	Cr	Cu	Mn	Ni	Zn
P0	2,150 ± 0,05 c	0,00 ± 0,00 c	41,400 ± 0,1 c	11,367 ± 0,153 a	7,200 ± 0,1 c	7,833 ± 0,058 c
P1	2,333 ± 0,10 c	0,011 ± 0,001c	42,630 ± 0,547 c	11,367 ± 0,153 a	7,430 ± 0,07 c	8,663 ± 0,360 b
P2	5,503 ± 0,18 a	3,633 ± 0,009 a	53,830 ± 1,041a	11,437 ± 0,163 a	9,433 ± 0,178a	9,227 ± 0,076 a
P3	4,563± 0,07 b	1,323 ± 0,067 b	44,833 ± 0,929b	11,397 ± 0,133 a	7,830 ± 0,286 b	8,963 ± 0,140 ab

Anyway, in carbons samples, the chromium contents were the lowest and the copper the highest. Metals contents are ranging between 0.011 $mg.kg^{-1}$ and 42.630 $mg.kg^{-1}$ for P1, 3.633 $mg.kg^{-1}$ and 53.830 $mg.kg^{-1}$ for P2 and between 1.323 $mg.kg^{-1}$ and 44.833 $mg.kg^{-1}$ for P3.

Except for zinc, metal content in carbon from water treatment of site P1, does not significantly difference to metal content in initial carbon (P0). This could be due to a higher content of zinc in the dam water. This result also showed that, apart from zinc, dam's waters contents in trace metallic elements are lowest than in initial carbon. This result supports our choice of water from this site as a witness.

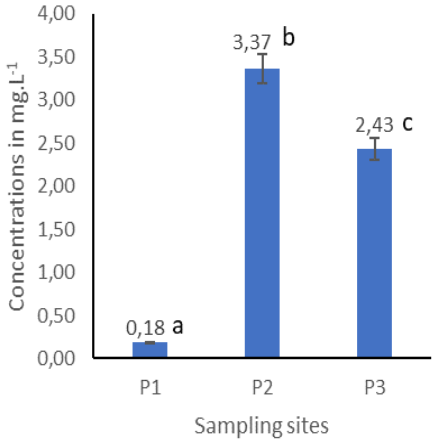
The carbons from water treatment at sites P2 and P3 have significantly higher metal concentrations than carbon P0. This shows that carbon have adsorbed metals from these waters. Indeed, in the literature, Singh, and Waziri, 2019 and Tan and al., 2017 [19, 20], have showed that coconut activated carbon has excellent adsorption capabilities of metals from either industrial wastewater or synthetic solutions.

Carbons used for water treatment of sites P2 and P3 are significantly rich in metals than those from water treatment of site P1. These results are due to a significant contribution of metals in waters of these sites. Indeed, it's well known that textile industry uses several dyes whose coloration is enhanced by the heavy metals they contain. As some of these dyes are not loosed during process, these metals are released into the wastewater of the textile industry [21, 22]. When wastewaters are directly released in environment or in rivers without any treatment this significantly contribute to enrich environment in metal and specially in TME.

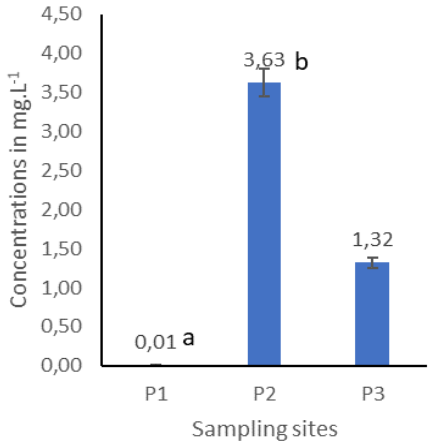
For this study, we used carbon with particle size lower than 0.25 mm with a specific surface area of 147.2 $m^2.g^{-1}$. Indeed, in literature it is reported that the adsorption of metals on activated carbon is better for a very fine particle size (lower than 0.25 mm) [23-25]. Moreover, previous studies have shown adsorptions of up to 99% on activated carbons or other adsorbents with specific surface areas between 3.48 and 537 $m^2.g^{-1}$ [26– 29]. With a specific surface area greater than 100 $m^2.g^{-1}$, such of carbon appears to be an interest adsorbent for trace metal elements analysis using X-ray fluorescence.

3.2. Contents of metallic trace elements in irrigation water

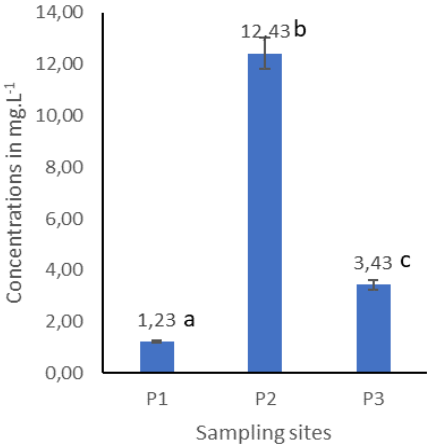
The determination of trace metal elements in water for watering crops is of paramount importance in the prevention of food poisoning. For example, cadmium is one of the most toxic heavy metals like lead and mercury. Their presence in drinking water, crops or food is generally liked to the agricultural itinerary or environmental pollution. This is the main reason for its monitoring in effluents, food and drinking water [30]. **Figure 3** below shows the concentration of trace metal elements in waters samples usually use for watering preurban cultures in study aera. **Figure 3(a)** shows cadmium concentration is water sample according to sites. It's content in waters samples is significantly different from one site to another and ranging between 0.18 $mg.L^{-1}$ (P1) and 3.37 $mg.L^{-1}$ (P2).



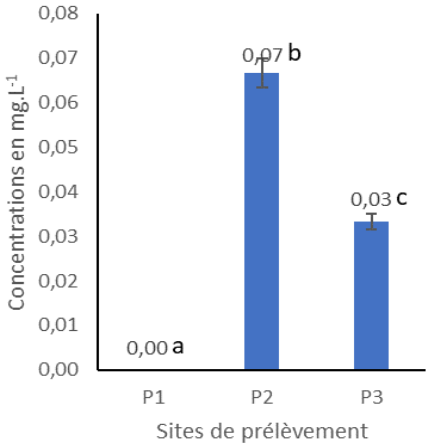
(a): cadmium concentrations



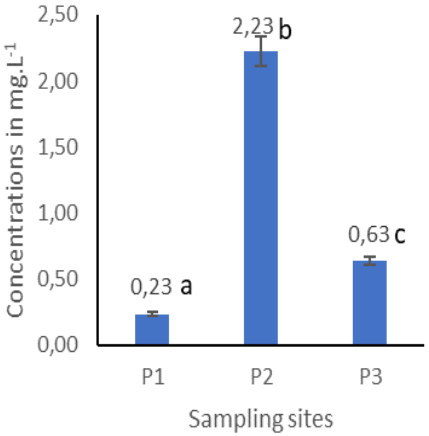
(b): chromium concentrations



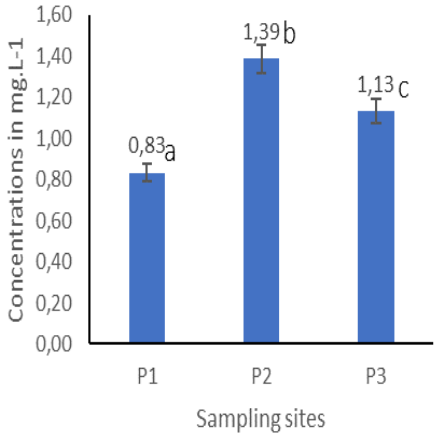
(c): copper concentrations



(d): manganese concentrations



(e) :nickel concentrations



(f) : Zinc concentrations

Figure 3: Concentrations of metallic trace elements in irrigation water

Waters from sites P2 and P3 have the highest contents compared to those of the control site P1. The high cadmium content of these waters may be linked to their industrial origin, as cadmium chloride is used in the dyeing and printing of textile designs [22], [31]. The cadmium values in all wastewater samples are above the FAO limit value of 0.05 $mg.L^{-1}$ [32]. Cadmium determined in water of control site (P1) and subsequently his value above the FAO limit suggest an anthropogenic origin probably environmental pollution linked to the use of the study area as discharge [33]. **Figure 3(b)** shows that chromium content in waters range from 0.01 $mg.L^{-1}$ (P1) to 3.63 $mg.L^{-1}$ (P2). Chromium values in water from P2 and P3 sites are well above 0.1 $mg.L^{-1}$, which is the limit value for chromium in agricultural wastewater [22, 34, 35]. Moreover, the chromium content of the water from these two sites (P2 and P3) is also higher than the chromium discharge standard of 0.5 $mg.L^{-1}$ set by Nigerian Ministerial Order No. 01164 of November 4, 2008, regulating discharges and emissions from facilities classified for environmental protection [36]. Although they are above the threshold values of the various standards, the concentrations of chromium in the water used for watering the P2 and P3 sites are lower than those established by Nwosu-Obieogu and Okolo in 2020 [37], which proved concentrations of around 200 $mg.L^{-1}$ in wastewater from textile industries in Nigeria. These high concentrations of chromium in wastewater from the textile industry are thought to be due to the use of chromium-based metal dyes in dyeing [23–25].

Concerning copper, **Figure 3(c)** shows that irrigation water has copper concentrations of 1.2 mg. L^{-1} in P1 and 12.4 $mg.L^{-1}$ in P2. Water from sites P1 and P3 have values less than 5 mg. L-1 which is the limit value recommended by the FAO in 2003 in its guidelines for the use of wastewater in irrigation. The copper contents of the waters of the P2 site are higher than the normative limit values and suggest a specific accumulation of copper in the waters of the P2 site linked to the fact that this is the discharge point for wastewater from the textile industry. Several authors state that phthalocyanines may contain copper in their structure [7, 26] and their use as dyes, could explain the presence of copper in the wastewater of the textile industry.

Regarding manganese, the concentrations presented in Figure 2d show that the waters at site P1 do not contain manganese. The P2 and P3 sites respectively have manganese contents of 0.07 mg. L^{-1} and 0.03 mg. L^{-1}. The P2 and P3 sites show values much lower than 0.2 mg. L^{-1} which is the limit manganese concentration authorized by the FAO in 2003 in its guide to the use of wastewater for irrigation [32]. Thus, in terms of the manganese concentrations, all the sites have water that can be used for crop irrigation.

Analysis of **Figure (3e)** shows that in the water nickel is present in concentrations between 0.23 mg. L^{-1} in P1 and 2.23 $mg.L^{-1}$ in P2. It can be seen that the concentrations at P2 and P3 sites are well above 0.2 $mg.L^{-1}$ which is the limit recommended by the FAO for the irrigation use of wastewater. Our results are significantly higher than those of Talouizte and al. in 2020 [5], who obtained concentrations between 0.01 and 0.02 $mg.L^{-1}$ in wastewater from the Moroccan textile industry. According to these authors, nickel as well as lead, copper, cadmium, and chromium are used to produce dyes used in textiles [5, 6]. However, the high nickel content could be linked to the fact that in Bouake the effluents are released into nature without any treatment. The high nickel values in the waters of P2 site would be due to its closer proximity to the textile industry compared to P3 site.

The presence of manganese and nickel in wastewater may also be due to an exchange between the soil and the slowly flowing wastewater. These results show that the textile industry does not contaminate the external environment with manganese and nickel.

Being among the most toxic elements, no trace of mercury and lead in the water was found, which confirms the absence of anthropogenic activities as sources of these metals in the study area [38, 39].

Zinc concentrations in waters ranged from 0.83 $mg.L^{-1}$ in P1 to 1.39 $mg.L^{-1}$ in P2. These values are below the limit concentration of zinc in waters for irrigation. According to the FAO, wastewater used for irrigation should not contain more than two (2) $mg.L^{-1}$ [32]. The presence of zinc in waters may be linked with the intensive use of fertilizers in urban agriculture. In fact, the water from sites P2 and P3, including surface runoff water as well as unprotected water reservoirs, could be contaminated during the spreading of fertilizers or by runoff from cultivated plots. In the literature, several authors claim that fertilizers are a source of soil and water contamination by metals such as zinc [31–33].

Conclusion

The objective of this work was to determine metal and specifically trace metal elements in water using activated carbon as adsorbing material and X-ray fluorescence spectrometry. It's have been showed that, X-ray fluorescence coupled with the use of activated carbon allowed to determine trace metallic elements in liquid sample such of water and to establish the metallic contaminants profile of the sample. This study also permits us to establish the potential origin of each metal found in waters samples.

However, the use of activated carbon as an adsorbent to measure the metal contents in liquid samples should be tested under several conditions before being used as a common analytical method. In addition, it is crucial to define the optimal adsorption conditions for proper use of this method as a rapid method of monitoring metals or metallic trace elements in water and to optimize the experimental models by statistical methods.

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