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# Experimental design for the optimization of preparation conditions of highly efficient activated carbon from *Glebionis coronaria* L. and heavy metals removal ability

*Hanane Tounsadi*[a,b], *Abderrahim Khalidi*[b], *Meryem Farnane*[a], *Mohamed Abdennouri*[a], *Noureddine Barka*[a,*]

[a] *Univ Hassan 1, Laboratoire des Sciences des Matériaux, des Milieux et de la Modélisation (LS3M), BP.145, 25000 Khouribga, Morocco*

[b] *Université Hassan II de Casablanca, Faculté des Sciences et Techniques, Laboratoire de Chimie Physique et de Chimie Bioorganique, BP 146, Mohammedia, Morocco*

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ABSTRACT

The objective of this study was the utilization of *Glebionis coronaria L.* biomass as a new precursor to produce highly efficient activated carbons by potassium hydroxide activation and their evaluation for heavy metals removal. The effects of four factors controlling the activation process, such as carbonization temperature (500–600 °C), activation temperature (400–500 °C), activation time (1–2 h) and impregnation ratio (g KOH/g carbon) (1–2) were investigated. To reduce the number of experiments, full factorial experimental design at two levels ($2^4$) was used to achieve optimal preparation conditions and better conditions for the removal of cadmium and cobalt ions from aqueous solutions. The experimental results showed that the activation time was the most significant factor with a positive impact for iodine number and a negative effect on methylene blue index. Further, and in same as, the interaction between activation time and impregnation ratio had a positive effect on iodine number and negative effect on methylene blue index. Therefore, the removal of cadmuim and cobalt ions onto activated carbons was more influenced by activation temperature and activation time with a negative effect. Although, the interaction between activation time and impregnation ratio was the most significant factor influencing the cadmium and cobalt ions removal. Based on the statistical data, the best conditions for the removal of cadmium and cobalt by *Glebionis coronaria* L. based activated carbons were indicated. Thus, the maximum iodine number and methylene blue index obtained under these experimental conditions were 752.69 mg/g and 284.04 mg/g respectively. Further, the optimized activated carbons were used in sorption isotherm. The maximum sorption capacities obtained with the application of the Langmuir isotherm model are 115.99, 106.93 mg/g for cadmium sorption and 44.85, 46.80 mg/g for cobalt sorption onto AC carbonized at 400 °C, activated at 500 °C during 1 h with an impregnation ratio of 2 g/g and AC pyrolized at 600 °C and activated at 500 °C for 1 h with an impregnation ratio of 2 g/g respectively. Those sorption efficiencies were shown greater than those of a commercial activated carbon used in water treatment.



* Corresponding author. Tel.: +212 661 66 66 22; fax: +212 523 49 03 54.
E-mail address: barkanoureddine@yahoo.fr (N. Barka).

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## 1. Introduction

Heavy metals are among the most important pollutants in aqueous media and industrial effluent. The main sources of heavy metals are the wastewaters from modern chemical industries such as metal plating facilities, fertilizer, mining, battery manufacturing, paper and pesticides, metallurgical, fossil fuel, tannery and production of different plastics such as polyvinyl chloride. During recent years, the rapid industrialization has strongly contributed to heavy metals release in to aquatic environment. Heavy metal species, especially cadmium and cobalt ions, have toxic effects on organisms and accumulate in biota. They are reported to be a source of major environmental and health hazards due to the unabated discharge of toxic effluents, their resistance to degradation, and adverse effects on both aquatic life and human consumption (Keshavarzi et al., 2015). Cadmium was the major priority pollutant; it was found to accumulate primarily in the kidneys and has a relatively long biological half-life in human bodies of 10–35 years (Hsanullah et al., 2015). Then, cobalt is related with the development of various respiratory and cardiovascular diseases. Also, it can caused a carcinogenic and genotoxic effect (Camara-Martos and Moreno-Rojas, 2016). For that, wastewater containing heavy metals needs to be purified and recycled in order to secure alternative sources of water.

A number of treatment technologies have been used for the removal of heavy metals from wastewater. The conventional methods that have been employed to remove the metal ions include electrochemical treatment (Thirugnanasambandham et al., 2015), reverse osmosis (Shen and Schäfer, 2014), flocculation (Lee et al., 2014), chemical precipitation (Al-Harahsheh et al., 2014), membrane filtration (Yuan and He, 2015), ion exchange (Guo et al., 2015) and adsorption (Tounsadi et al., 2015). Among these various methods, adsorption is universal method for treating heavy metals with efficiency and low expense. Several adsorbents natural or synthetic have been developed. Subsequently, many research studies established that carbon-based adsorbent proved to be the most cost-effective for the sorption of heavy metals from wastewater (Ali, 2010).

Activated carbon can be produced by both naturally material and synthetic of carbonaceous solid precursor (Girgis et al., 2002). It has been classed based on its starting material. The nature of starting material or precursor plays a leading role in influencing the quality, characteristics and properties of the resulting activated carbon (Abechi et al., 2013). In addition, the properties of the resulting activated carbon will also be influenced by types of activating reagents (Hirunpraditkoon et al., 2011), activation time (Gliscinska and Babel, 2013), impregnation conditions, carbonization temperature (Yahya et al., 2015), and others. For that, experimental designs have been used to control the different factors which influence and interfere in the preparation, in order to optimize experimental conditions.

Recently, the activated carbon was produced by a variety of raw materials from vegetables. Also, the abundance and availability of biomasses assure us using them as a precursor for production of activated carbon. Therefore, a number of lignocellulosic biomasses including *Diplotaxis* Harra (Tounsadi et al., 2016), waste potato peels (Kyzas et al., 2016a), rice husk (Sugashini and Begum, 2015), pine tree and coconut shell (Tonucci et al., 2015), Pennisetum alopecuroides (Kang et al., 2016), Moso bamboo (Huang et al., 2015), Zizania caduciflora (Liu et al., 2014a), apricot stone (Abbas et al., 2014a), Salvadora persica (Wahid et al., 2013), Moringa pods (Zaini et al., 2014), Jatropha wood and Peanut shell (Gueye et al., 2014) have been employed extensively as a new precursor in the production of activated carbon for the removal of a number of heavy metals from water.

There are two different processes for activated carbon preparation; chemical and physical treatment. Both treatments are responsible in varying the shapes and the sizes. The manufacture of activated carbon by physical activation requires high temperatures (800–1000 °C), which involves high power consumption and a low yield of carbon (Nowicki et al., 2015). In contrast, in the chemical activation, the carbonization temperature is ranged between 400 and 600 °C. On the other hand, in chemical treatment, precursors are impregnated by an activating reagent and followed by heating process. Among chemical activating agents widely used such as NaOH, KOH, $K_2CO_3$, $HNO_3$, $ZnCl_2$ or $H_3PO_4$. The chemical method needs lower temperatures, produces higher yield and creates well developed microporosities. Depending on the conditions of the manufacturing process, the typical surface areas for activated carbon vary from 500 to 1400 $m^2/g$, although values as high as 2500 $m^2/g$ have been reported (Liu et al., 2014b).

The aim of this study was the preparation of activated carbons from *Glebionis coronaria* L. (ACs) by potassium hydroxide activation. Then, the optimization of the preparation conditions and the removal of cadmium and cobalt ions using a factorial experimental design. Further, the factors included in the experimental design were the carbonization temperature, activation temperature, activation time and impregnation ratio. Four responses are analyzed, which are; iodine number (IN), methylene blue index (MB index), cadmium and cobalt ions removal (Cd(II), Co(II)). To investigate the optimal conditions for the production of ACs, and to establish the removal of heavy metals, a $2^4$ full factorial experimental design was used. The ACs produced at the optimal conditions was characterized by scanning electron microscopy (SEM), BET surface area and the functional groups were quantified by Boehm titration. Also, for the optimized activated carbons, the equilibrium data of heavy metal ions sorption were studied by Langmuir and Freundlich isotherm models.

## 2. Materials and methods

### 2.1. Materials

All the chemicals used in this study were of analytical grade. $Cd(NO_3)_2.4H_2O$ (98%), $Co(NO_3)_2{\cdot}6H_2O$ (98%), potassium hydroxide (KOH) (98%), iodine ($I_2$), sodium thiosulfate ($Na_2S_2O_3{\cdot}5H_2O$), $Na_2CO_3$, $NaHCO_3$, HCl (37%) and commercial activated charcoal (powder form) were purchased from Sigma–ldrich (Germany). $HNO_3$ (65%) was provided from Sharlau (Spain). NaOH from Merck (Germany), potassium iodide (KI) was obtained from Pharmac (Morocco) and methylene blue ($C_{16}H_{18}ClN_3S$) (85%) was purchased from Panreac (Spain).

### 2.2. Preparation of activated carbons

*G. coronaria* L. was collected from the region of Khouribga, Morocco. The steams of the plant was crushed and sieved. Fraction with particle size of 1–2 mm was selected for the preparation of activated carbons. The biomass was carbonized at 500 or 600 °C in a tubular furnace during 2 h. After that, the furnace was switched off and cooled down to 25 °C. Carbonized samples were impregnated with potassium hydroxide at KOH