### Chapter 3

# Design of Experiments for Evaluation of Variables Involved in the Removal of Heavy Metal Ions From Water Using Agro-Industrial Waste-Based Adsorbents

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#### **ABSTRACT**

The design of experiments (DOE) is a useful tool to define the most significative variables of a process, and the optimal operation conditions, or reduce the noise caused by uncontrollable variables. An advantage of the employ of DOE is the reduction of time required for analysis and costs associated. The Factorial, surface response methodology, and Taguchi have been employed to analyze the variables involved in the removal of heavy metal ions coming from wastewater by adsorption employing low-cost adsorbents, which include agro-industrial waste and biomass. The most important variables associated to increase of adsorption capacity and evaluated by researchers include temperature, pH, adsorbent dose, initial concentration of metal, and particle size.

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#### INTRODUCTION

The industrial activity historically has caused an increase in the concentration of heavy metal ions in water associated with the incorporation of wastewater into the environment (Barakat, 2011). Heavy metal ions are toxic and half of these, when are emitted into the environment, are a risk factor to human health, animals and ecosystems (Wu et al., 2019). Most studied heavy metal ions in water are cobalt (Co), iron (Fe) lead (Pb), cadmium (Cd), copper (Cu), nickel (Ni), hexavalent chromium Cr(VI) and arsenic (As) (Fu & Wang, 2011). Different techniques have been employed for the removal of heavy metals, such as precipitation, adsorption, ion exchange, and membrane filtration as reverse osmosis.

Low-cost adsorbents are obtained from agro-industrial wastes as raw materials, such as rice husk, nutshell, wood, coconut shell, bean hulls, artichoke, seed, banana pith, eggshell, crimp husk and tree leaves, the employ of these can help to reduce environmental pollution. An advantage associated with the use of low-cost adsorbents for heavy metal removal is that at the end of metal recovery, the used biomass can be filtered of an aqueous solution, dried and burned for energy generation. A disadvantage associated with the use of low-cost adsorbents can be related to low removal capacity, however, the use of designs of experiments (DOE) is a useful tool that has been employed to define the experimental parameters, their levels, and interactions that represent a high contribution in adsorption processes for heavy metals removal. The DOE allows reducing the analysis time and costs with a lower number of better-designed trials.

Some types of DOE employed for the improvement of the heavy metal ions removal process include factorial design (full factorial and fractional factorial), Taguchi design, response surface methodology; which also considers Box-Behnken, and central composite design. The type of DOE is generally selected by researchers in order to take advantage of the sorbents derived from abundant renewable resources, agro-industrial by-products or the waste plant material which are an economic and environmentally friendly source of raw material.

The present chapter is focused on the application of experiments design for the determination of optimal conditions of controllable factors and their influence on the removal of heavy metal ions employing low-cost adsorbents.

#### BACKGROUND

#### **Heavy Metal Pollution**

Currently, there is a growing interest in environmental care. The industrial activity is responsible for the increase in the concentration of heavy metal into the environment. There is not a clear definition of heavy metals, however, the term heavy metals is often used to name metals and metalloids characterized by a density that exceeds 5 g·cm<sup>-3</sup>, most of them are highly water soluble, present high toxicity when accumulating in living organisms and ecosystems and some of these have been identified as carcinogenic agents. The heavy metal ions definition includes those that represent a threat associated with exposure, however, some of these are essential elements for the growth, reproduction and/or survival of living organisms (Wu et al, 2019). The heavy metal most studied are: Hg, Pb, Cd, Cu, Ni, Cr and As, being the last a metalloid usually classified as a heavy metal (Fu & Wang, 2011).

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