Chapter 2
Design of Experiments to Optimize Soxhlet–HTP Method to Establish Environmental Diagnostics of Polluted Soil:
Optimization of the Soxhlet–HTP Method by DOE

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ABSTRACT

One of the great challenges of the environmental diagnoses of soils contaminated with hydrocarbons is the optimization of analytical determinations. For this reason, this chapter evaluates the extraction of hydrocarbons by the Soxhlet method through the design of experiments (DOE), variating three different solvents, three soils, and three extraction times. Soil was experimentally contaminated at different concentrations, and hydrocarbons totals relying on conditions organic matter, electrical conductivity,

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pH and textures, amount of sample, solvents, and NaSO4, were studied. The variables were evaluated by means of an analysis of the Taguchi design and a factorial design, with the results the significant and optimal parameters of the process were determined, which were solvent type and time (10 hours and dielectric constant of 9). Also, the model discards the soil properties. These results will save time and resources, and they reduce errors.

INTRODUCTION

During the last decade, Mexico has been involved in some major changes, mainly focused in the public policies, for example, recently the Mexican government enacted a law to reform the constitution, this law consisted on the allowance of private capital investment in the energy sector in order to maximize the use of our natural resources, also to improve the economy of our country. After that, institutions such as the National Hydrocarbons Commission (CNH), and the Safety Energy and Environment Agency (ASEA) were formed. Currently, these organizations regulate the activities of the petroleum industry and determine the mechanisms for evaluating the impacts that they have and will have on our ecological systems (Hernández, Santillan, Sabelkin, and Parra, 2003; Barrientos & Añorve, 2014, Merchand, 2015; Martínez-Lara & Páez Melo, 2017).

Within the regulations that were established by these newly created public agencies it is specified that analyzes and studies performed should be based on scientific studies, an example is the environmental-base-lines made for reopening or development in the hydrocarbons sector, which specify that the proposed analytical methods must be supported by rigorous scientific studies, mainly, if these are not regulated at least they should be validated, this is the reason why some tools such as design experiments (DOE) and experimental mathematical models have a high application and potential, including some that have already been successfully tested (Morales-Bautista, Méndez-Olán, Hernández-Jiménez, and Adams, 2018).

Currently, one of the great challenges of environmental analytical chemistry is the complexity of the behavior of analytes in the different matrices, in the case of soils, there are still few basic studies in tropical areas that have been subject to constant oil spills, many of them, have been restored but some of the areas with agricultural use vocation continue with problems to establish crops and have resulted in negative impacts in the primary sector, leading to legal and social conflicts for many decades, especially in the southeast states (Castro, Acevedo-Berruecos, Urbieta, Iturbe and, Delgado-Rodríguez, 2012; Adams, Álvarez-Ovando, and Castañón, 2015). In these sites, residual hydrocarbons associated with those effects have been identified, some studies mention that there is a different effect between each type of soil, since these can vary from one site to another and, therefore, have different capacity to respond to a similar pollutant (Zamora, Ramos y Arias, 2012; Palma-Cruz, Pérez-Vargas, Casado, Guzmán, and Calva-Calva, 2016).

Different studies have shown that omitting this parameter causes high uncertainties in declared environmental diagnoses, besides recently was found that the type of hydrocarbon can also influence the level of affectation, in general, it was observed that depending on the fraction of hydrocarbon present in the environment is the type of remediation to be used (García-López, Zavala-Cruz, and Palma-López,