Tunning Nb_2O_5 nanoparticles' specific surface area by a machine learning approach for application in heterogeneous photocatalysis

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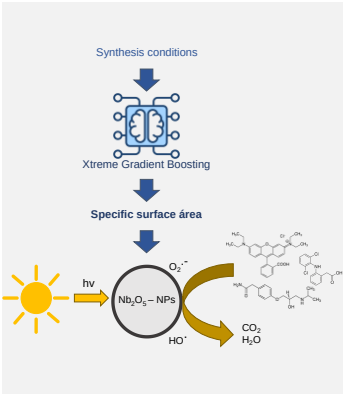

Niobium(V) oxide nanoparticles (Nb_2O_5-NPs) have been applied in the treatment of wastewater by heterogeneous photocatalysis, due to their textural and structural properties, associated with considerable photocatalytic activity. Furthermore, the use of computational tools based on machine learning appears as an attractive alternative in elucidating the mechanisms involved in the photodegradation of emerging organic pollutants. In this context, the present work aims to evaluate the application of a Machine Learning (ML) algorithm (Xtreme Gradient Boosting – XGB) to predict the specific surface area (SSA) of Nb_2O_5-NPs. The XGB algorithm presented the best performance with a maximum tree depth of 4m, achieving coefficients of determination of 0.9613 and 0.9259 for training and testing, respectively. Therefore, the development of the ML study showed promising potential to be used before the experimental steps to reduce the time and costs associated and tune the Nb_2O_5-NPs SSA.

Introduction

Access to clean and safe water is one of the challenges nowadays due to incorrect discharge and/or inefficiency of conventional treatments of wastewater containing emerging organic pollutants (EOPs) [1]. Thus, advanced treatment processes have been studied and applied to remove these organic pollutants, such as catalytic processes. Heterogeneous photocatalysis stands out for its versatility and the possibility of using semiconductors (catalysts) with regeneration capacity, which when photoactivated promote a series of oxidation-reduction reactions onto the catalytic surface, generating hydroxyl radicals (●OH) responsible for removing EOPs. [2].

Among the catalysts, niobium(V) oxide nanoparticles (Nb_2O_5-NPs) present thermal and chemical stability, unique Lewis and Bronsted acid sites, low toxicity, and bang gap energy in the range of ~3.1 – 3.4 eV, which is suitable for redox reactions in the photodegradation OEP [3]. The synthesis of Nb_2O_5-NPs can be achieved by several preparation conditions, including route, type of precursor, and temperature. These conditions will affect the Nb_2O_5-NPs properties such as crystalline structure, morphology, and specific surface area. Among these properties, the specific surface area (SSA) is important due to the availability of the active sites under the surface for redox reactions [4].

In this context, Machine Learning (ML) studies can assist the tunning of Nb_2O_5-NPs SSA for further applications in heterogeneous photocatalysis. The use of ML algorithms is helpful for identifying patterns, predicting responses, identifying main variables from a database, and reducing the time and costs associated with experimental steps [5]. In this context, the present work aims to evaluate the application of a ML study in the prediction of the SSA values of Nb_2O_5-NPs for heterogeneous photocatalysis.

Material and Methods

The database used in the ML study was collected, filtered, and selected from the Scopus platform (www.scopus.com) using the Boolean logic with the descriptors “niobium(V) oxide” AND “nanoparticles” AND “photocatalysis”. The main input variables were the type of precursor, synthesis route, and temperature. The output variable was the SSA values. The database was divided into training (80%) and testing (20%), and the input data was normalized according to Eq. 1 [6].

$$x_{in} = \gamma \frac{x_i - x_{min}}{x_{max} - x_{min}} - \beta\gamma \quad (1)$$

Where x_{in} is the normalized value, x_i is the raw data of the input variable, x_{min} is the minimum value for the input variable, and x_{max} is the maximum value for the input variable. The values of the γ and β coefficients used were, respectively, 2 and 0.5.

The Extreme Gradient Boosting (XGB) algorithm was used for the ML study of Nb_2O_5-NPs SSA in Python 3.8 (Google Colaboratory). The booster *dart* was used with a step size shrinkage (*eta*) of 0.2 and the maximum depth of a tree was evaluated in the range of 1 – 100 m. The performance parameters used for the XGB algorithm were the coefficient of determination (R^2) and the square root of the mean square error (*RMSE*).

Results and Discussion

Exploratory analysis

The database was composed of 188 experimental data organized in 47 lines per 4 columns. Figure 1 shows the results of the data survey. The main routes of synthesis were sol-gel (64 %), hydrothermal (28 %), and heat degradation (9 %), according to Figure 1(a). The precursors often used in the synthesis of Nb_2O_5-NPs were niobium ammonium oxalate (53 %), niobium pentaethoxide (30 %), and niobium chloride (17 %) according to Figure 1(b). The greater frequency of the sol-gel route is attributed to its control of the size and morphology of metallic oxide nanoparticles [7]. The

niobium ammonium oxalate presented greater frequency due to its solubility in water compared to the other precursors, which favors the synthesis of Nb_2O_5-NPs. The range of temperatures used in the synthesis heat treatments was in the 100 – 750 °C and the Nb_2O_5-NPs SSA values were in the 4.44 – 302 $m^2.g^{-1}$ range.

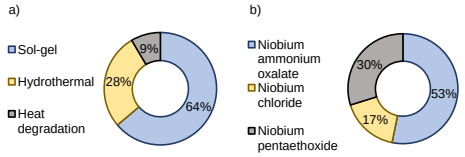

Figure 1. (a) Main synthesis routes and (b) metal precursors for the exploratory analysis of the Nb_2O_5-NPs.

Machine learning

The maximum depth of a decision tree used in the XGB algorithm was evaluated to determine the optimal depth and avoid data overfitting. Figure 2 shows the evolution of the performance parameters (R^2 and *RMSE*) in function of the maximum tree depth.

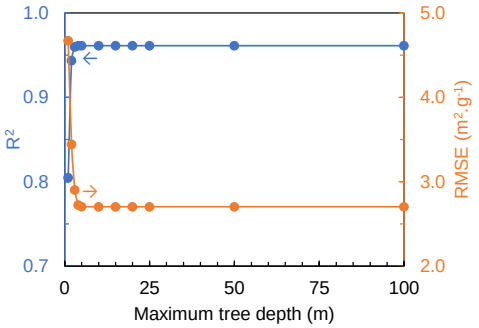

Figure 2. Performance of the XGB algorithm as function of the maximum tree depth.

As presented in Figure 2, above 4 m there are no improvements in the performance of the XGB algorithm in the prediction of the Nb_2O_5-NPs SSA values. Thus, the training and testing of the XGB algorithm was performed using a 4 m decision tree. The R^2 obtained for the training and testing was 0.9613 and 0.9259, respectively. The *RMSE* for training and testing was 2.744 $m^2.g^{-1}$ and 3.857 $m^2.g^{-1}$. Moreover, the performance parameters showed the quality of the correlation obtained by the XGB algorithm. Figure 3 illustrates the correlation between the values predicted by the XGB algorithm as well as the observed values of specific surface area (training and testing).

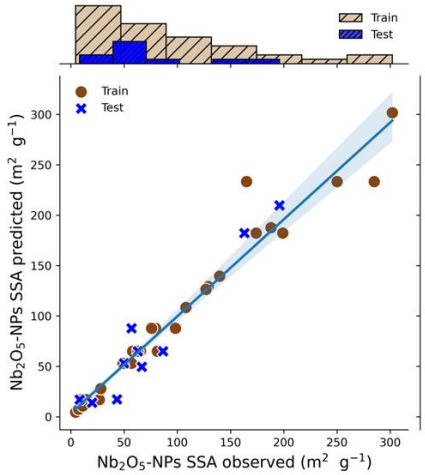

Figure 3. Correlation between predicted and observed values of the SSA for Nb_2O_5-NPs.

Most of the observed values are within the prediction range of the carried out ML study. Thus, the use of ML computational techniques through the XGB was able to predict the specific surface area values of Nb_2O_5-NPs based on information about the synthesis parameters.

Conclusions

The evaluation of the SSA for Nb_2O_5-NPs was possible by application of a ML study. The XGB algorithm showed optimal performance with a maximum depth tree of 4 m, with coefficients of determination above 0.90 for training and testing. Therefore, the use of ML computational studies proved to be a promising tool for predicting experimental values of Nb_2O_5-NPs SSA for application in heterogeneous photocatalysis.

Acknowledgments

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