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Use of design of experiments to optimize the synthesis of heterogeneous catalysts supported on activated carbon starting from ornamental stone residue

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In 2023, Brazil exported 1.82 Mt of ornamental stones, with Espírito Santo being responsible for around 75 % of this amount,[1] showing the importance of this sector for the economy of this Brazilian state.[2] However, the steps to obtain the finished product generate around 30 to 40 % of solid waste.[3] This work aims to explore the use of activated carbon to disperse CaO and MgO particles derived from ornamental stone waste, with the produced composite materials being employed as catalysts in the transesterification of soybean oil with methanol to produce biodiesel. The synthesis of the heterogeneous catalysts was optimized using experimental design methodology. A screening stage was conducted to assess the significance of the studied variables, including the mass ratio between stone waste and activated carbon, the temperature and the residence time of the heat treatments used to prepare the catalysts. For this study, a 2^3 + CP (central point) full factorial design was employed, with two levels for each variable and triplicate CPs, resulting in a total of 11 experiments.[4] The response variable was the conversion of soybean oil into biodiesel, which was monitored and quantified by solution ^{1}H nuclear magnetic resonance (NMR) spectroscopy. The catalysts were prepared by wet mixing; in this route, the activated carbon and the ornamental stone waste are first added to a 3.75 mol/L NaOH solution and the mixture is subsequently heat treated at different temperatures and times.[5] Within the experimental domain of the full factorial design, the highest biodiesel conversion value was 84.6 %. The experimental conditions corresponding to the highest conversion were: mass ratio of stone waste : activated carbon : NaOH = 50 : 20 : 30; heat treatment temperature = 800 °C; residence time = 1 h. Therefore, the experimental design methodology shows that the heat treatment at 800 ºC for 1 h is required to obtain catalysts with good activity, showing that the chosen route is promising for the production of carbon-supported heterogeneous catalysts starting from ornamental stone residues and an environmentally-friendly activated carbon.

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