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ÁREA: Catalysts

USE OF MURUMURU SEED SHELLS AS PRECURSORS FOR SULFONATED BIOCHARS IN VANILLIN SYNTHESIS

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Resumo-Abstract

The Amazon region has a vast diversity of trees and plants, offering valuable biological resources. Among them, murumuru stands out, especially for the cosmetic industry, due to its excellent active ingredients. The industry uses the pulp and kernels of murumuru, but the seed shells are generally discarded as waste. To explore a new application for this agro-industrial waste, murumuru shells were utilized as biomass for the production of biochars, which served as support for heterogeneous catalysts. The biochars from murumuru shells (BCM) were sulfonated using 3-mercaptopropyltrimethoxysilane (MTPS) and concentrated sulfuric acid to produce two catalysts with sulfonic groups: BCM-MTPS and BCM-SULF. These catalysts were tested in reactions for the synthesis of vanillin, a flavoring widely used in various industries. The acid-base titration to evaluate the acidic sites revealed that the BCM-MTPS catalyst had a surface acidity of 0.97 mmol H^+ g^{-1}, while BCM-SULF had 2.29 mmol H^+ g^{-1}. These data suggest that the functionalization of BCM-MTPS may not have been efficient, as its surface acidity index is lower than that of the original biochar, which had 1.24 mmol H^+ g^{-1}. EDS analysis indicated that both catalysts have a typical composition of carbonaceous materials. However, no sulfur traces were detected in BCM-MTPS, suggesting the absence of sulfonic groups. In contrast, BCM-SULF showed a sulfur concentration of 5%. Catalytic tests, involving the oxidation of vanillyl alcohol to vanillin, were conducted for 1 hour under reflux at 80°C. The GC-MS results of the reaction without the catalyst showed a 34.5% conversion of vanillyl alcohol, the precursor reagent. For the BCM-MTPS and BCM-SULF catalysts, this conversion was 18.7% and 100%, respectively. Due to its high efficiency, BCM-SULF was subjected to 3 reuse reactions, and in the last reaction, it achieved approximately 80% conversion of the precursor. The vanillin conversion, the desired product, for BCM-MTPS and BCM-SULF was 8.8% and 11.9%, respectively. However, it was observed that in subsequent reactions with BCM-SULF, despite the loss of catalyst efficiency, vanillin conversion increased, reaching a maximum of 46.8% in the second reuse and 31.1% in the final cycle. This effect may be due to vanillin overoxidation, a phenomenon described in the literature as the further conversion of vanillin into other compounds. It is concluded that BCM-SULF showed better catalytic performance compared to BCM-MTPS. Therefore, the synthesis steps of BCM-MTPS should be reevaluated, and further characterizations and catalytic tests with BCM-SULF are recommended to improve vanillin conversion. This demonstrates that BCM-SULF is a promising catalyst for vanillin synthesis and other reactions requiring acid catalysis.

Keywords: Murumuru seed shells. Heterogeneous catalyst. Biochar. Sulfonation. Vanillin.

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