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Synthesis and Characterization of CaO Nanoparticles Impregnated in KIT-6 for Application in Moringa Oil Cracking.

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Abstract

The growing demand for renewable fuels is driven by the need to ensure energy security and environmental balance. Among the promising alternatives is the production of biohydrocarbons from vegetable oils and animal fats. Processes such as catalytic pyrolysis and catalytic cracking stand out for their efficiency, especially when using suitable catalysts. In this context, KIT-6 is a promising material due to its high surface area and porous structure, which facilitates the diffusion of reactants in catalytic reactions. However, because it lacks active sites, modification with metal oxides, such as calcium, is necessary to optimize its catalytic performance. This study aimed to synthesize and characterize calcium oxide (CaO) nanoparticles, impregnate them into KIT-6, and apply them in the catalytic cracking of moringa oil using thermogravimetric analysis. The synthesis of KIT-6 followed the method described by Kleitz, while the synthesis of CaO nanoparticles was performed by slowly mixing NaNO3 (0.2 mol/L) and NaOH (0.4 mol/L) solutions. The resulting precipitate was filtered, washed, dried, and calcined at 600°C for 6 hours. The incorporation of calcium into KIT-6 was performed using the solvent excess method, with 5% (wt%) calcium derived from the previously synthesized oxide. The synthesized materials were characterized by thermogravimetric analysis (TGA), X-ray diffraction (XRD), X-ray fluorescence (XRF), Fourier-transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM). The catalytic activity of the materials was evaluated in the cracking of moringa oil, using 10 mg of oil and 10% (wt%) catalyst between 30-800°C, with a thermobalance. The thermogravimetric curves for KIT-6 and CaO-KIT-6 exhibited two mass losses at lower temperatures, associated with the removal of physically and chemically adsorbed water, while smaller variations between 270°C and 900°C were attributed to the dehydroxylation of silanol groups. CaO showed discrete mass losses between 340-421°C, corresponding to the decomposition of $Ca(OH)_2$ and between 510-640°C, related to $CaCO_3$, both formed during synthesis or storage. Low-angle XRD patterns of KIT-6 and CaO-KIT-6 displayed Miller indices (211), (220), and (332), confirming the presence of an ordered cubic structure even after calcium insertion. High-angle diffractograms showed characteristic peaks of CaO and calcium hydroxide, corroborating TGA data. XRF analysis indicated 93.2% purity for CaO (corroborating TG and FTIR results) and 5.5% calcium content in CaO-KIT-6, confirming expected impregnation values. The FTIR spectra of CaO exhibited bands related to O-H stretching and C-O and O-C-O deformation, confirming the presence of $Ca(OH)_2$ and $CaCO_3$. SEM micrographs of KIT-6 and CaO-KIT-6 samples showed cubic morphology on a nanometric scale, while CaO exhibited spherical agglomerates of varying nanometric sizes. In the cracking, the moringa oil, KIT-6 and CaO-KIT-6 showed a single event with maximum temperatures at 419°C, 420°C and 416°C, respectively, indicating the formation of compounds at lower temperatures for CaO-KIT-6 compared to thermal cracking and pure KIT-6. The process using CaO showed four mass losses over different temperature ranges, indicating the formation of different compounds, which may be related to the material's purity (93.2%) and its morphology, which facilitated the cracking of oil molecules into various fractions. Based on the results, it can be concluded that the synthesis and characterization of CaO nanoparticles and their incorporation into KIT-6 preserved the mesoporous structure. TGA, XRD, and XRF analyses confirmed the material's purity. CaO and CaO-KIT-6 catalysts exhibited superior performance compared to thermal cracking and pure KIT-6, with more thermal events and reduced reaction temperatures. The results suggest that these catalysts have significant potential for biomass conversion into renewable biofuels.

Keywords: Catalysis, Biofuels, Moringa Oil, Calcium Oxide

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