Synthesis of $\text{Zr}_6\text{Nb}_2\text{O}_{17}$ mixed oxide for use as a heterogeneous catalyst support.

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Abstract: In this research, potassium catalysts supported in mixed oxide $\text{Zr}_6\text{Nb}_2\text{O}_{17}$ were prepared. They were used in transesterification reactions of ethanol and soybean oil for biodiesel production. The support was prepared by precipitation of hydrated zirconia with the addition of $\text{ZrOCl}_2$ solution in ammonia. The solid was filtered, washed, dried at 383K, and then it was impregnated for 24h, under stirring, in a peroxoniobium (V) solution. The peroxoniobium (V) solution was prepared by adding $\text{NbO(NH}_4\text{)}(\text{C}_2\text{O}_4)_2$, under stirring, to a 30% $\text{H}_2\text{O}_2$ solution acidified with $\text{HNO}_3$ concentrated solution at pH~0.5, and then the solution was heated at 323K for 1h. After impregnation, the solid was dried by a gentle evaporation and then it was calcined at 623K for 2h and at 873K for 2h in order to form the mixed oxide [1]. The mixed oxide structure was confirmed by X-Ray Diffraction analysis. Then the support was impregnated with a KOH solution. The catalysts were dried at 383K and then they were calcined at 473K for 24h to activation. The transesterification reactions were done following the factorial design with four variables: time of reaction, mole ratio of ethanol:oil, amount of catalyst, and catalyst KOH content. The temperature was maintained constant at 358K and the reaction was held under stirring in a round bottom flask with a reflux system. The reaction products were purified. The conversion rate of the products was obtained by Nuclear Magnetic Resonance Spectroscopy analysis. Conversions were more than 77% under the experiments conditions: conversions more than 99% were obtained in all reactions with the biggest mole ratio of ethanol:oil, and with the biggest amount of catalyst. In another conditions with the increase of the time, the conversion was increased from 77% to 80%.

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