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Asymmetric Michael-addition catalyzed by a cinchona alkaloid derivative non-covalently immobilized over layered materials

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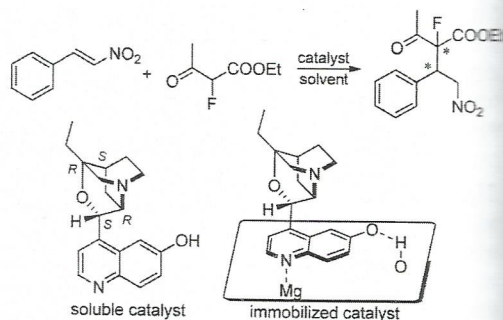
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Preparation of optically pure chiral intermediates by catalytic asymmetric methods is in the forefront of the modern chemical research. The application of environmentally benign and sustainable processes in the production of chiral fine chemicals requires the development of highly efficient, recyclable, heterogeneous chiral catalysts. Such materials may be obtained either by surface modification of solid catalysts or by immobilization of homogeneous stereoselective catalysts. Immobilization of recently developed chiral organocatalysts is a promising method to obtain chiral materials for applications in a large variety of asymmetric reactions, such as enantioselective Michael-additions. However, the simplicity of the immobilization procedure is of paramount importance in determining the applicability of the catalyst. Our aim was to develop immobilized chiral organocatalysts by a simple procedure for catalyzing the asymmetric addition of an α -substituted β -ketoester to nitrostyrene.

Initially, we prepared a chiral organocatalyst, i.e. β -isocupreidine, easily obtained in a one-step procedure from the natural cinchona alkaloid quinidine. This derivative was similarly efficient as the compound reported in the literature used to catalyze the chosen test reaction, shown in the figure below [1]. The cinchona derivative was immobilized by simple adsorption on the surface of a commercial layered double hydroxide under basic conditions, without significant intercalation between the anion-exchanger lamellae. The material was characterized by its powder X-ray diffraction pattern and by IR spectroscopy.

The results obtained with the immobilized chiral catalysts were close to that reached under homogeneous reaction conditions. In an appropriate, i.e. apolar solvent, the solid catalyst could be recycled up to eight times without losing its catalytic activity and with only small decrease in the enantioselectivity. The IR spectrum of the spent catalyst confirmed that the cinchona alkaloid remained adsorbed on the solid surface. The catalyst was also used in a continuous-flow system affording sustained conversion. The developed chiral solid is a novel, highly efficient, easily obtainable immobilized cinchona alkaloid appropriate for the preparation of valuable chiral intermediates.



Asymmetric Michael-addition used as the test reaction.

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References

- [1] H. Li, S. Zhang, C. Yu, X. Song, W. Wang, Chem. Commun. (2009) 2136.