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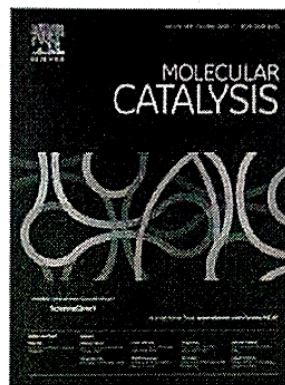
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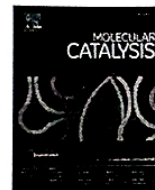
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Research Paper

Catalytic Baeyer-Villiger oxidation of cyclohexanone over magnesium oxide-zinc oxide clusters supported on porous silica microspheres

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ABSTRACT

ϵ -Caprolactone is an essential monomer for the synthesis of poly-caprolactone and is primarily produced via the Baeyer-Villiger oxidation of cyclohexanone. This study focuses on the preparation of $\text{MgZn}_x\text{-PSM}_y$ catalysts by loading Mg and Zn onto porous silica microspheres for the hydrogen peroxide oxidation of cyclohexanone to ϵ -caprolactone. The metal oxides exist in the form of clusters. The catalysts were characterized using X-ray diffraction (XRD), scanning electron microscopy (SEM), nitrogen adsorption-desorption analysis, Fourier transform infrared spectroscopy (FTIR), Raman spectroscopy, ultraviolet-visible spectroscopy (UV-Vis), and X-ray photoelectron spectroscopy (XPS) to confirm their successful synthesis. The effects of catalyst preparation method, metal molar ratios, metal-to-support mass ratios, catalyst dosage, reaction time, reaction temperature, and hydrogen peroxide concentration on catalytic performance were systematically investigated, along with the reusability of the catalyst. Under optimized conditions, with an Mg:Zn molar ratio of 3:1, a metal-to-porous silica microsphere mass ratio of 0.21, a catalyst loading of 5 % (relative to the mass of cyclohexanone), a reaction temperature of 80 °C, and a reaction time of 10 hours, the cyclohexanone conversion reached 91 %, with an ϵ -caprolactone selectivity of 93 % and a yield of 84 %.

1. Introduction

Polymer materials have significantly enhanced the diversity and convenience of daily life. With societal advancements, their applications in both production and daily activities have expanded considerably. However, commonly used polymers such as polypropylene (PP), polyethylene (PE), polystyrene (PS), and polyvinyl chloride (PVC) are non-degradable. Once discarded, these materials persist in the environment, posing severe threats to ecosystems, including soils, oceans, lakes, and forests. To mitigate these environmental impacts, the development and adoption of degradable polymer materials are imperative. Among degradable polymers, polycaprolactone (PCL) stands out as a biodegradable polyester with exceptional mechanical properties and biocompatibility. Its versatility has made it widely applicable in areas such as food packaging, drug delivery [1], tissue engineering [2], and wound healing [3]. PCL is typically synthesized via the ring-opening polymerization of ϵ -caprolactone, which is primarily produced through the Baeyer-Villiger oxidation of cyclohexanone.

In 1899, Adolf Baeyer and Victor Villiger first described the oxidation of menthone to its corresponding lactone using peroxysulfates and

concentrated sulfuric acid [4]. Since then, the Baeyer-Villiger (BV) oxidation has become a cornerstone transformation in organic synthesis, with ϵ -caprolactone being a key monomer produced via the BV oxidation of cyclohexanone. Traditional BV oxidation employs organic peracids as oxidants, such as meta-chloroperoxybenzoic acid, peracetic acid, and trifluoroperacetic acid. These peracids exhibit strong oxidizing capabilities and achieve high conversion rates without requiring a catalyst. However, they are expensive, impact-sensitive, unstable, and associated with significant safety risks. Their production relies on hazardous concentrated hydrogen peroxide, which complicates transportation and storage. Moreover, peracid-based oxidations generate equivalent amounts of organic waste acids or acid salts, which are corrosive to equipment and may trigger secondary reactions, reducing selectivity. These drawbacks, along with environmental pollution concerns, underscore the need for safer, more stable, and environmentally friendly oxidants to replace traditional peracid systems.

Alternative oxidants include molecular oxygen and hydrogen peroxide. Molecular oxygen has weak oxidation ability and cannot directly generate oxygen radicals to convert ketones into esters. Typically, an aldehyde is required as a sacrificial agent, such as

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