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Toward an Inherently Safer Alternative for Operating *N*-oxidation of Alkylpyridines: Effect of *N*-oxide on Lutidine – Water Phase Separation

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HIGHLIGHTS

- Effect of 2,6-lutidine *N*-oxide on 2,6-lutidine/water phase separation is studied
- 2,6-lutidine *N*-oxide has a positive influence on the mixing of the system
- Homogeneous mixtures result if *N*-oxide above 20% (w/w) in ternary system at 110°C
- Inherent safety concept – “hybridization” to *N*-oxidation reactions identified

ABSTRACT

The *N*-oxidation of alkylpyridines is an industrially important reaction since it produces alkylpyridine *N*-oxides that are pharmaceutical intermediates. The aqueous hydrogen peroxide used to oxidize the alkylpyridine has a tendency to decompose during the reaction thereby introducing serious hazards for the process. The decomposition is accelerated during the *N*-oxidation of higher order alkylpyridines (lutidines, collidines) due to mass transfer limitations caused by the separation of the liquid into organic and aqueous phase. Also, the presence of phosphotungstic acid (catalyst) in the aqueous phase further intensifies the peroxide decomposition reducing the safety and efficiency of the process. The current work investigates the influence of the product *N*-oxide on the mixing between alkylpyridine and water, which is primarily responsible for the liquid phase heterogeneity during the *N*-oxidation. Ternary mixtures of 2,6-lutidine, 2,6-

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