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Abstract: Herein, we developed the first photochemical reaction set-up under solid-state conditions using a resonant acoustic mixer (RAM), a novel mechanochemical methodology that eliminates the need for milling media. This innovation outperforms previous approaches in the ball mill under avoidance of milling balls and their related disadvantages. Further, it enables the facile performance and up-scaling of photochemical reactions up to a factor of ten. We investigate the influences of liquid and solid additives on the rheology and performance of two model reactions and demonstrate and compare the efficiency and applicability of two custom-designed photoreactors.

Introduction

Light is one of the most environment-friendly activation modes, being both renewable and waste-free. However, conventional photochemical reactions suffer from sustainability issues, as they are often performed in highly diluted solutions due to the poor penetration depth of photons which results in substantial waste production.^[1,2] Owing to the global incentives towards environmentally friendly synthesis alternatives, research on solvent-free pathways is increasing.^[3-6] Nonetheless, typical photochemical approaches in the solid-state are performed on glass plates suffering from poor diffusion and requiring small sample batches. Mechanochemistry is an alternative solid-state synthesis strategy capable of overcoming these disadvantages. Mechanochemical synthesis describes the performance of chemical reactions under the impact of mechanical energy avoiding bulk solvents,^[5-8] providing greener and more efficient reactions,^[7-12] and also opening the pathways towards otherwise inaccessible products.^[13,14] Next to the limited amount of publications on combining photochemistry and ball milling,^[15-22] our working group has recently addressed the challenge by designing translucent quartz glass milling vials, opening up the opportunities for photochemical reactions with higher energy UVC light.^[23-26] This innovation has overcome a major hurdle that enables efficient photochemical organic transformations without the use of metal catalysts and bulk solvents.^[17,24] While these milling vessels are suitable for use with soft milling media made from polymers, quartz glass is not resistant to harder materials. In mechanochemistry, the usage of ball mills often

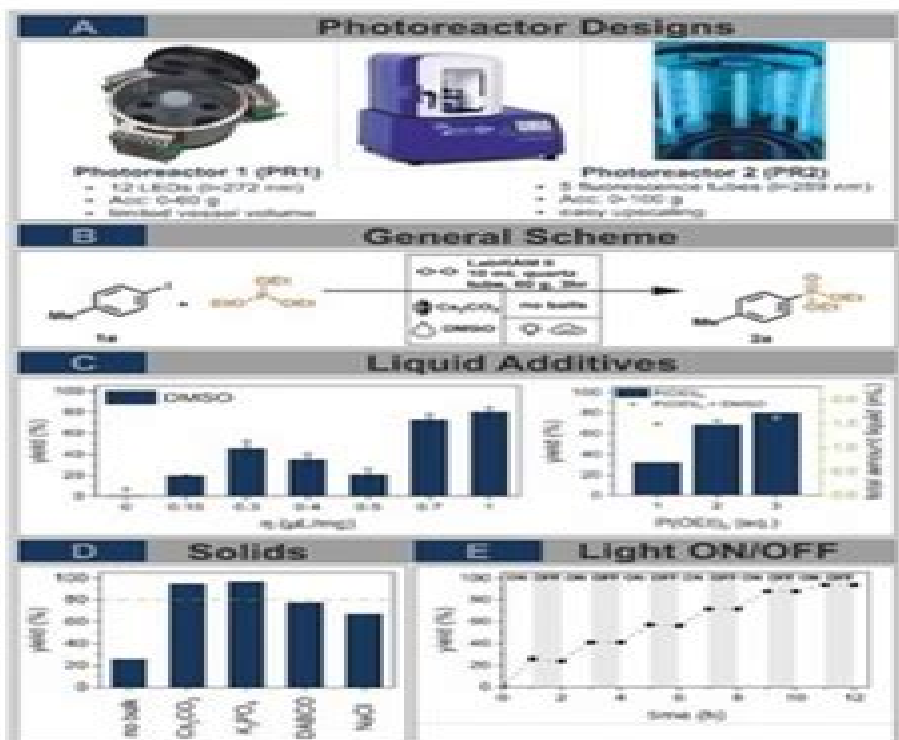


Figure 1. A: Photoreactor designs for the resonant acoustic mixer (RAM, middle), right (PR1) based on LED arrays, left (PR2) based on low-pressure mercury fluorescence tubes. B: General scheme of model reaction, photochemical phosphonation reaction of 4-iodotoluene (1a) with triethyl phosphite, reaction conditions if not stated otherwise: 0.38 mmol 1a, 3 eq. Pr(OEt)₃, 2 eq. Cs₂CO₃, 1 g SiO₂, and DMSO ($\eta=0.3$) were mixed in a 1.4 mL quartz glass tube at 60 g in the RAM for 3 h under ambient air conditions and constant irradiation of 12 UVC-LEDs ($\lambda=272$ nm) in PR1. C: Investigation of the influence of liquid additives including, left: amount (n) of DMSO and right equivalents of Pr(OEt)₃ in dependency of the total amount of liquid. D: Influence of solid additives, green line indicates the yield under previously optimized conditions. E: Kinetic investigation of the phosphonation reaction of 4-iodotoluene (1eq.) under optimized conditions using 3eq. Pr(OEt)₃, 1.2 g K₂PO₄, DMSO ($\eta=1$) were mixed under ambient air conditions in the RAM at 60 g for 1 h under irradiation using PR1 followed by 1 h in the dark. The process was repeated until full conversion of the substrate to the product was achieved. Corresponding conversions were determined by ¹H-NMR spectroscopy by analyzing a small amount of the crude reaction mixture.

Solid State Photochemistry Monographs In Modern Chemistry 8

V. Ramamurthy, Yoshihisa Inoue



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