# Process intensification for the direct synthesis of adipic acid in a micro packed bed reactor

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| Abstract title | PROCESS INTENSIFICATION FOR THE DIRECT SYNTHESIS OF ADIPIC ACID IN A MICRO PACKED BED REACTOR |
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Background: Most industrial processes to produce adipic acid include two steps[1], i.e., an aerobic oxidation of cyclohexane to form K/A oil and followed by a nitric oxidation to obtain adipic acid. During this process, a large amount of $N_2O$ is produced as a by-product.

$H_2O_2$ is a “green” oxidant since water is the sole byproduct[2] and has a high oxygen content. So many researchers have started to use $H_2O_2$ to oxidize cyclohexene directly to produce adipic acid[3]. To the best of our knowledge, however, all reports concerning the adipic acid synthesis from cyclohexene and $H_2O_2$ were carried out in a laboratory batch reactor, which need long reaction times and is not easy to scale up.

Aim: The focus of this project is on the transformation of the reaction from batch reactor to micro flow reactor, which should reduce the reaction time by at least an order of magnitude and overcome scale-up limitations.

Method: The synthesis of adipic acid starting from cyclohexene and $H_2O_2$ was carried out in a micro packed bed reactor. The reaction uses $Na_2WO_4 \cdot 2H_2O$ as a catalyst and $[CH_3(n\text{-}C_8H_{17})_3N]HSO_4$ as a phase transfer catalyst[4] without additional solvent. A parametric process optimization was done and the multi-injection of $H_2O_2$ was explored.

Results: It is found that 50% wt $H_2O_2$ is much more productive than 30% wt $H_2O_2$ under the same condition. We found that the acid plays an important role in the direct synthesis of adipic acid. Under given conditions, an sulfuric acid concentration 0.63 mol/l is found to give the highest isolated yield of adipic acid. Based on the concept of Novel Process Windows[5], the influence of elevated temperatures is investigated and 100℃ is shown to be the optimum temperature for the reaction, which results in almost 50% isolated yield of adipic acid when the residence time is 20 min (50% $H_2O_2$: cyclohexene: $Na_2WO_4 \cdot 2H_2O$: $[CH_3(n\text{-}C_8H_{17})_3N]HSO_4$ molar ratio is 440: 100: 6: 6). The decomposition of $H_2O_2$ was also studied and the result shows that the concentration of $H_2O_2$ decreased a lot even without reaction under 100℃.

Conclusion: Compared to soybean oil epoxidation, which we investigate as well [6], generic learning was made – such as the even higher exothermicity and mass-transfer limitations (need for phase-transfer catalyst) given for the direct synthesis of adipic acid; common to both and to overcome is the deleterious $H_2O_2$ decomposition (needing $H_2O_2$ excess or lowering yield).

[1] Davis D.D. In Ullman’s Encyclopedia of Industrial Chemistry, 5th ed.; Gerhartz W., Ed., John Wiley and Sons: New York, 1985, Vol. A1, pp 270.

[2] Catalytic Oxidations with Hydrogen Peroxide as Oxidant, Ed. G. Strukul, Kluwer Academic, Dordrecht, 1992.

[3] (a) Sato K., Aoki M. and Noyori R., Science 281 (1998) 1646; (b) Lapisardi G., et al., Catal. Commun. 5 (2004) 277.

[4] Sato K., Noyori R., et al. J. Org. Chem. 61 (1996) 8310.

[5] Hessel V., Chem. Eng. Technol. 32 (2009) 1655.

[6] Cortese B., Croon M.H.J.M. de and Hessel V., Ind. Eng. Chem. Res. 51 (2012) 1680.

**Images**

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