Accelerated Discovery and Characterization of Nanoscale-Covalent Organic Frameworks for Photocatalytic Water Splitting

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1. Introduction

Imine-based covalent organic frameworks (COFs) that can easily form extended conjugated structures with good visible-light absorption range have been widely used in photocatalysis.[1] However, designing COFs with suitable structure for photocatalytic processes takes time, due to the large number combinations of possible monomer combinations. The development of high-throughput synthetic methods could greatly increase the number of materials being synthesized and tested[2]. However, the synthesize process of bulk COFs usually involves high temperature and pressure, which is not easily achievable in a high-throughput workflow. The emergence of nanoscale COFs (nanoCOFs) synthesized by a simple surfactant-assisted process under room temperature and normal pressure provides an opportunity to develop an automated synthesis.[3] Herein we, have developed the high-throughput synthesis of nanoCOFs using a Chemspeed platform. To evaluate reaction success a computer vision-based screening method was established using the reaction colour change(ΔE) and laser intensity (predict particle size) (Fig.1). The successfully synthesized nanoCOFs were then evaluated for their sacrificial HER performance.

2. Practical note

For reproducible nanoCOF synthesis, we use a Chemspeed Swing platform to standardize liquid mixing and reaction timing. Images for color analysis were collected using a Logitech C930e 1080p webcam and analyzed in the CIELAB color space. Laser scattering images and particle size estimation were performed as previously reported.[4]

3. Substantial section

Here we report the accelerated discovery of nano-COF photocatalysts based on a combinatorial library of 3 aldehydes and 10 amines, using automated synthesis to prepare 30 possible nanoCOFs. By applying color change and light-scattering thresholds, nanoCOFs with extended light absorption range and nanoscale particle size were identified for photocatalytic testing. This method achieved 90% accuracy (2 false positives) in identifying nanoCOF formation when compared to the results from standard characterization methods (UV-vis spectroscopy and FT-IR spectroscopy). Out of the 30 possible aldehyde/amine combinations, 15 successfully formed nanoCOFs. Integrating this synthetic route with the automated workflow developed for photocatalytic performance testing enabled an efficient photocatalyst discovery workflow to be achieved.[5]

3.1 Related work

Prior computer-vision (CV) reaction monitoring has mainly been demonstrated as a general tool for extracting kinetic or endpoint information from videos in high-throughput experimentation, but not as a materials-specific surrogate characterization method for nanoparticle-forming frameworks[6,7].
Therefore, we automate nanoCOF synthesis and establish a CV-based characterization that uses color evaluation and light-scattering pattern changes of nanoCOF colloids as a rapid, in-situ proxy to substitute or triage traditional ex-situ characterization for high-throughput decision making[8]; this is enabled by the ability of the nanoCOFs to form stable colloids in solution.

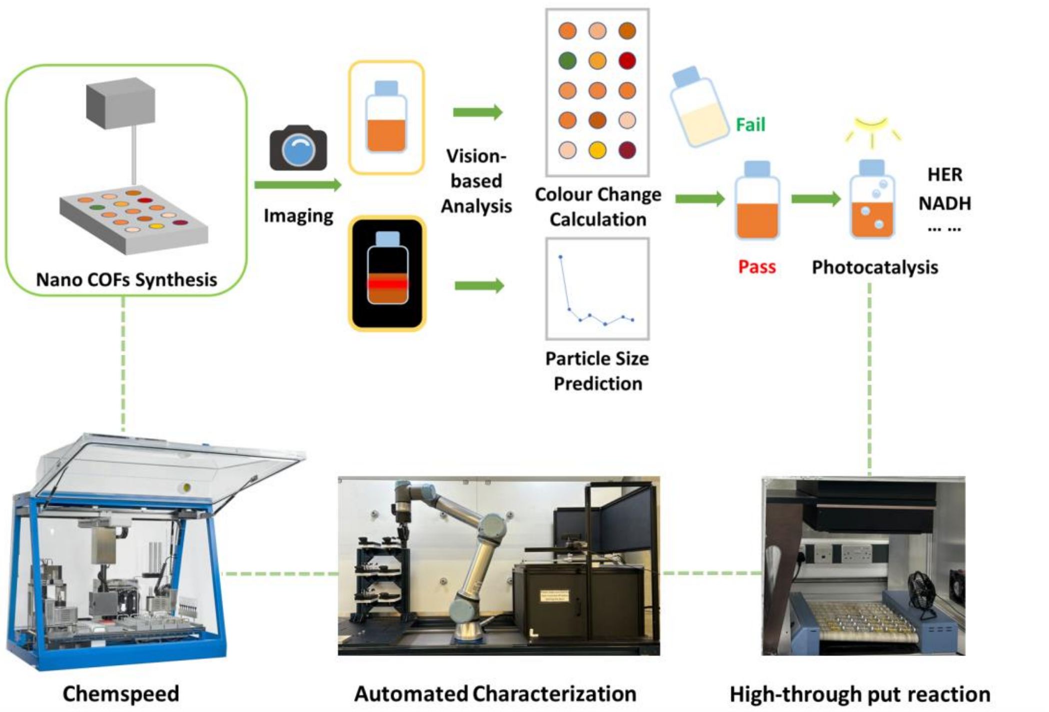

Fig. 1: Workflow of synthesis, characterization and discovery of nanoCOFs for photocatalytic sacrificial HER.

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