Supporting Information

Continuous Synthesis of 2,5-Hexanedione through Direct C-C Coupling of Acetone in a Hilbert Fractal Photo Microreactor

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S1. Light transmittance of FEP tubing

The FEP tubing used in this work represented a wall thickness of 0.5 mm. Since the FEP film with an equal thickness was not available, we determined the light transmission of a FEP film with 0.45 mm thickness using UV-vis2700, as shown in Fig. S1. It can be found that the transmittance was 90-95% at wavelength >400 nm, and it drops quickly when the wavelength <400 nm. Therefore, it can be deduced that the FEP tubing would cut off partial transmission of Hg lamp.

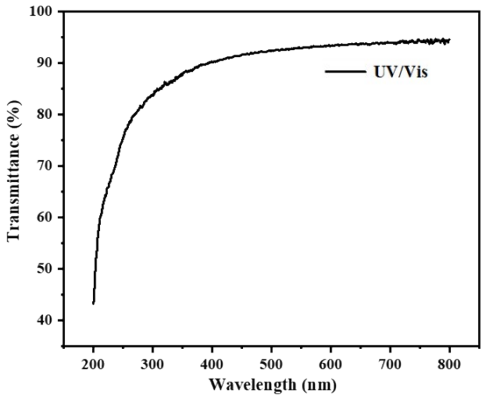

Fig. S1. Light Transmittance of FEP film with a thickness of 0.45 mm

S2. Product analysis by GC-MS

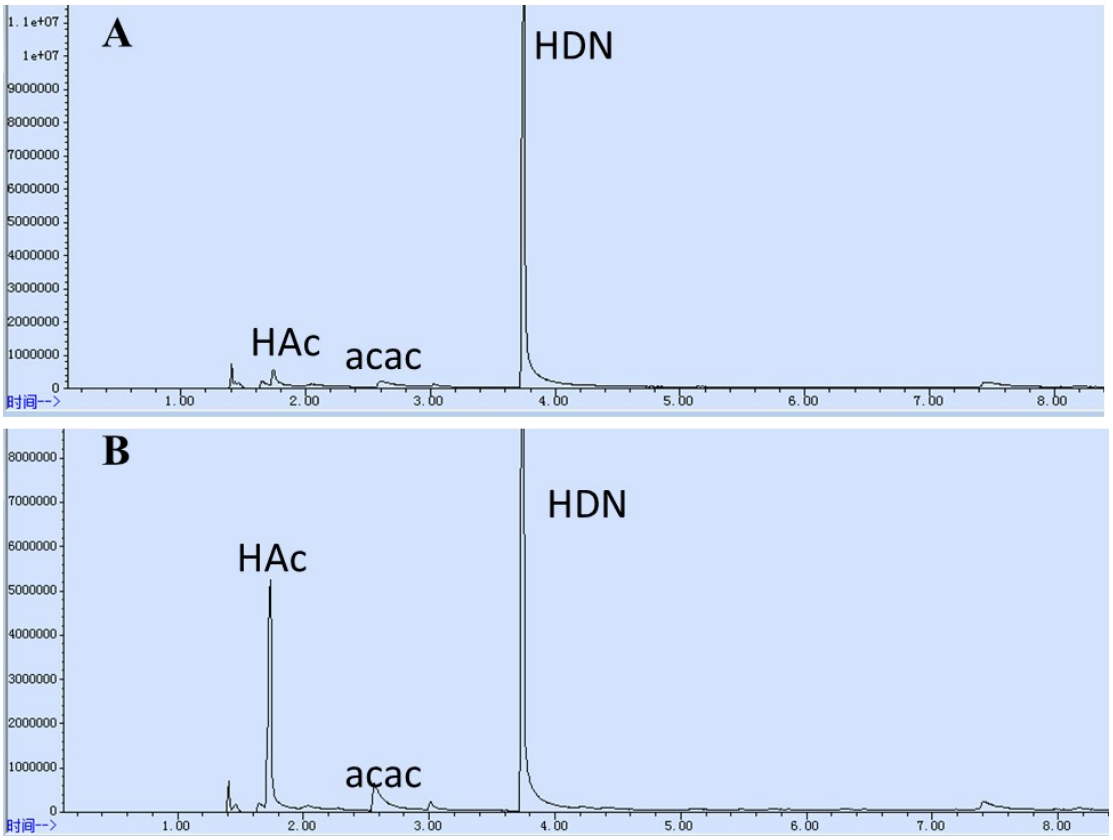

Fig. S2. The GC spectrum for the continuous synthesis of 2,5-hexadione in Hilbert fractal PMR with 2% H_2O_2, (A) 2.5 min residence time, (B) 15 min residence time

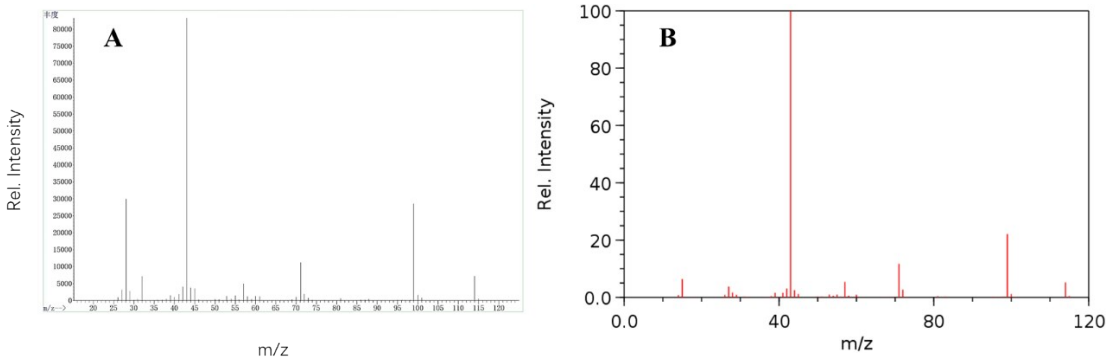

Fig. S3. (A) The mass spectrum of main product in the reaction system, (B) the standard mass spectrum of 2,5-HDN.

S2. Apparent quantum yield (AQY) determination

The apparent quantum yield (AQY) was determined by equation as follows:

$$AQY(\%) = \frac{2 \times N(HDN)}{N(photons)} \times 100 \quad \text{(S1)}$$

where N(HDN) and N(photons) denote the number of produced HDN molecules and the incident photons, respectively.

$$N(HDN) = Conc.(HDN) \times {}^{V_r}\!/_{\tau} \times 6.022 \times 10^{23} \quad \text{(S2)}$$

$$N(photons) = \frac{P \times t \times \lambda}{h \times c} \quad \text{(S3)}$$

where t is the reaction time, λ is the wavelength of the incident light, h is the Planck constant, c is the velocity of light and P is the energy flux of incident light, which can be determined using equation as follows:

$$P = E \times S \quad \text{(S4)}$$

where E denotes the irradiance of incident light and S is the area of the reactant exposed to light.

In the present work, the wavelength λ and irradiance E of the used high-pressure Hg lamp were measured by an UV spectral irradiance colorimeter (OHSP-350UV, Hopocolor). Same distance (10 cm) between the colorimeter and the Hg lamp was kept as that applied in the actual reaction. As shown in Fig. S2, the high-pressure Hg lamp represented a wide range of wavelength, and the dominant wave was at 366 nm. Thus, this value was used for our calculation of AQY. Moreover, since the Hg lamp was supplied by alternating current (AC), the irradiance E was found to fluctuate. Therefore, the mean value of 310 mW/cm^2 of five measurements was applied here for the AQY estimation.

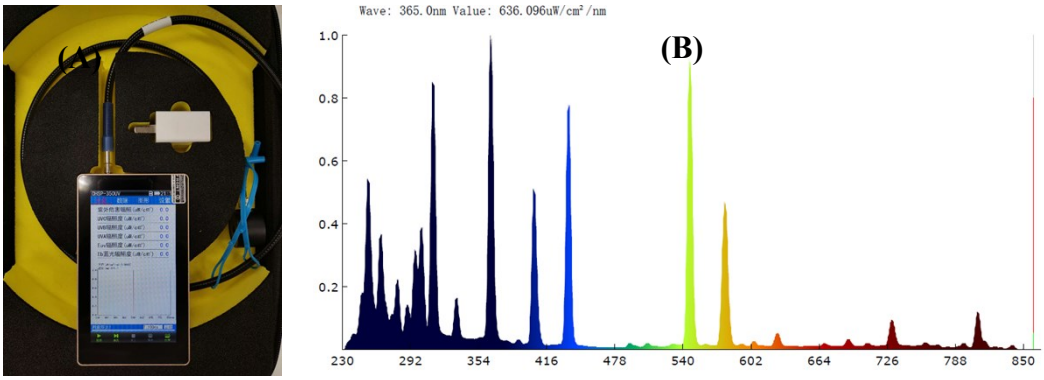

Fig. S4. (A) Photo of used UV spectral irradiance colorimeter, (B) Emission spectra of 300 W Hg lamp at a distance of 10 cm

The microchannel in Hilbert fractal PMR represented a depth of 1mm, a width of 1mm and a length of 700 mm. Therefore, the total area of the reactant exposed to the light S was 7 cm^2. For the FEP capillary PMR, it represented an inner diameter of 1 mm, and a length of 2.55 m. The area of the reactants exposed to light was estimated as follows:

$S = 0.5 \times 0.5 \times \pi \times d \times l = 0.5 \times 0.5 \times \pi \times 1 \times 2550 = \ 2000\ mm^2 = 20\ cm^2$

With the above information, the AQY could thus be calculated using eq S1.

S3. Experimental results

Table S1. Products selectivities in both PMRs with 2% H_2O_2 volume ratio

Res. T.	Selectivity Hilbert PMR			Selectivity FEP capillary		
min	HDN	HAc	acac	HDN	HAc	acac
2.5	97.2%	2.4%	0.4%	96.0%	3.7%	0.3%
5	92.0%	5.8%	2.2%	91.8%	5.8%	2.4%
7.5	86.5%	10.9%	2.6%	85.6%	9.9%	4.5%
10	80.5%	14.3%	5.2%	80.0%	14.6%	5.4%
15	80.1%	14.3%	5.6%	79.9%	14.3%	5.8%

Table S2. Products selectivities in both PMRs with 3% H_2O_2 volume ratio

Res. T.	Selectivity Hilbert PMR			Selectivity FEP capillary		
min	HDN	HAc	acac	HDN	HAc	acac
2.5	93.2%	6.4%	0.3%	89.5%	10.1%	0.5%
5	85.7%	12.0%	2.3%	85.2%	12.3%	2.5%
7.5	78.7%	16.9%	4.5%	78.8%	16.3%	4.8%
10	75.9%	18.3%	5.8%	74.6%	19.4%	6.0%
15	73.3%	19.4%	7.3%	72.4%	20.6%	7.0%

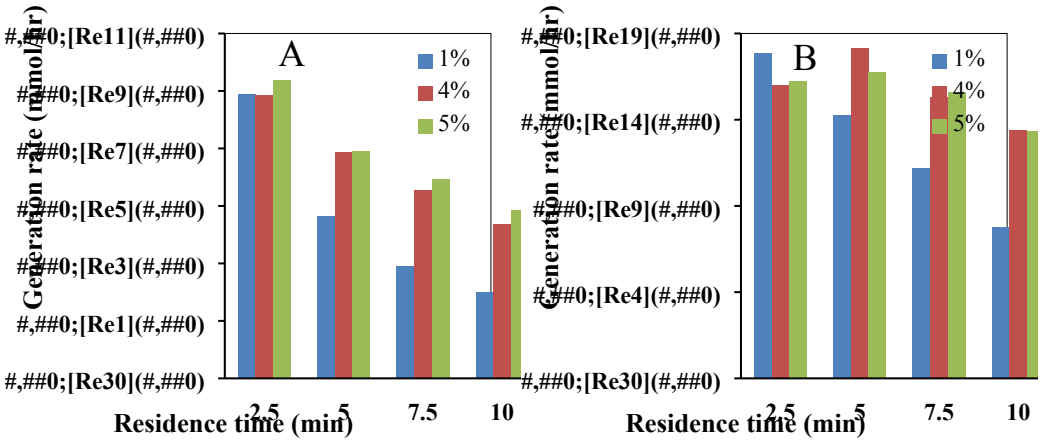

Fig. S5. Generation rate of 2,5-HDN in Hilbert fractal PMR (A) and FEP capillary PMR (B) with 1%, 4% and 5% H_2O_2 solution

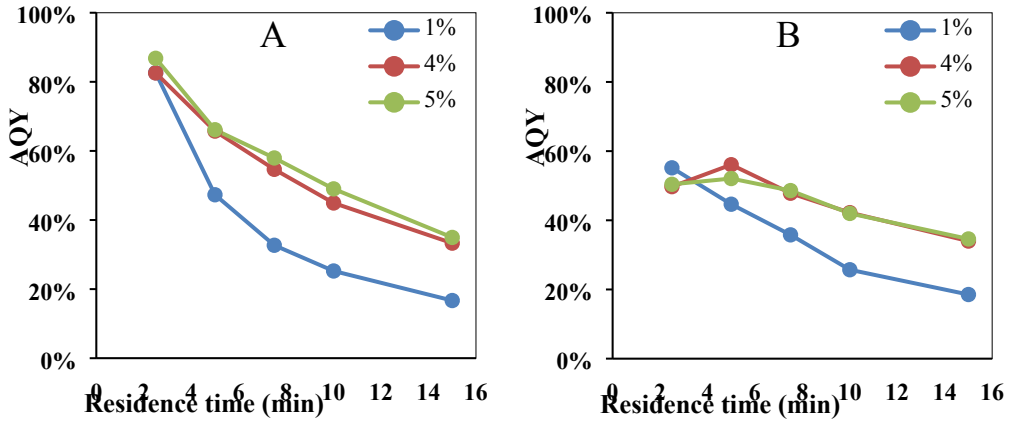

Fig. S6. AQY of Hilbert fractal PMR (A) and FEP capillary PMR (B) with 1%, 4% and 5% H_2O_2 solution