Supplementary Information

Test of Cobalt leaching

The leached Co\textsuperscript{2+} ion concentration was measured by inductively coupled plasma (ICP, Leeman Teledyne) which determines metal ions in a solution for identification and differentiation of metal ions down to ppb levels. Solutions were obtained after the reactions, filtered, and mixed with 2% nitric acid aqueous solution before analysis.

The cobalt concentration leached from the reaction solution after filtration by microfiltration membrane (0.22 μm) was tested by ICP in the current study. The cobalt leachate testing by ICP is illustrated in Fig. S1. The cobalt leachate is hardly any cobalt leachate. The Cobalt cations concentration after catalytic process were below 0.03mg/L, which is much less than the LD50 value of soluble cobalt salts. The leached cobalt concentration was very low because of the Cobalt leaching from the catalyst is limited and a low catalyst concentration (10 mg Co\textsubscript{3}O\textsubscript{4}/GO in 100 mL solution) was used in the reaction. So, the AOPs-SRs can’t cause secondary pollution.

Fig. S1 Dissolution of Co from the catalysts after degradation reaction process. (a) uncalcined Co\textsubscript{3}O\textsubscript{4}/GO; (b) calcined at 300 °C; (c) calcined at 500 °C; (d) calcined at
700 °C; (e) calcined at 900 °C. (Reaction conditions: [Orange II] = 0.6 mM, [PMS] = 6 mM, [catalyst] = 0.05 g/L, pH = 7.0, and \( T = 25 \) °C).

The adsorptive property of the catalysts

In order to test the adsorptive property of the catalysts, the degradation reaction was conducted with catalyst alone and without PMS. All experiments were conducted in neutral conditions with the catalyst dosage of 0.05 g/L and the pH during the entire reaction process was almost constant. Fig. S2 shown the adsorption of Orange II onto \( \text{Co}_3\text{O}_4/\text{GO} \) uncalcined and calcined at 300 °C, 500 °C, 700 °C, and 900 °C, respectively. The adsorption/desorption were found to be a reversible process, the concentration of Orange II only decreased around 10% in the first few minutes and then a plateau was reached. Since a negligible extent of dye removal occurred without PMS, the small drop in Orange II concentration could be due to the physical adsorption on the catalysts surface.

![Fig. S2 The adsorptive property of the catalysts.](image)

BET surface areas of the catalyst
The BET surface areas were measured by a BET analyzer (BK122T–B, JWGB, China) using the liquid nitrogen adsorption method. 

The surface area measurement of the Co$_3$O$_4$/GO uncalcined and calcined at 300 °C, 500 °C, 700 °C, and 900 °C via nitrogen gas absorption yielded a BET value of 126.7, 123.6, 127.9, 129.2, and 130.4 m$^2$/g. When the calcination temperature was increased, the change of the BET value is small. Generally, for the metal oxide, the higher the temperature employed in the calcinations process, the lower the surface area and the larger the particle size. However, high-temperature calcination results in the exfoliation of GO and a decrease in oxygen-containing groups on the surface of GO, which lead to the formation of reduced GO (rGO) or even graphene. This phenomenon may result in the increase of the BET surface areas of the catalyst. So, with the increase of calcination temperature, the BET value increases a little. Therefore, neither the adsorption of Orange II onto the catalysts nor the small increase of BET value will be responsible for the activity of the catalysts.

**Degradation curves of Orange II in the dark**

The degradation reaction experiments were performed in the dark at the same condition. The Degradation curves of Orange II by using Co$_3$O$_4$/GO uncalcined and calcined at 300 °C, 500 °C, 700 °C, and 900 °C as a catalyst in the dark reaction environment were shown in Fig. S3. The trend of degradation reaction in the dark is identical with the normal conditions. The results indicated that there is no contribution of photo-catalysis in the process.
Fig. S3 Degradation curves of Orange II by using Co$_3$O$_4$/GO uncalcined and calcined at 300 °C, 500 °C, 700 °C, and 900 °C as a catalyst. Inset: In the dark reaction environment at the same condition.
Supplementary Figure Captions

**Fig. S1** Dissolution of Co from the catalysts after degradation reaction process. (a) uncalcined Co$_3$O$_4$/GO; (b) calcined at 300 °C; (c) calcined at 500 °C; (d) calcined at 700 °C; (e) calcined at 900 °C. (Reaction conditions: [Orange II] = 0.6 mM, [PMS] = 6 mM, [catalyst] = 0.05 g/L, pH = 7.0, and T = 25 °C)

**Fig. S2** The adsorptive property of the catalysts.

**Fig. S3** Degradation curves of Orange II by using Co$_3$O$_4$/GO uncalcined and calcined at 300 °C, 500 °C, 700 °C, and 900 °C as a catalyst. Inset: In the dark reaction environment at the same condition.