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## The 5th International Symposium on Environmental Economy and Technology



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## Degradation mechanism of Direct Pink 12B treated by iron-carbon micro-electrolysis and Fenton reaction

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### Abstract

The Direct Pink 12B dye was treated by iron-carbon micro-electrolysis (ICME) and Fenton oxidation. The degradation pathway of Direct Pink 12B dye was inferred by ultraviolet visible (UV-Vis), infrared absorption spectrum (IR) and high performance liquid chromatography-mass spectrometry (HPLC-MS). The major reason of decolorization was that the conjugate structure was disrupted in the iron-carbon micro-electrolysis (ICME) process. However, the dye was not degraded completely because benzene rings and naphthalene rings were not broken. In the Fenton oxidation process, the azo bond groups surrounded by higher electron cloud density were first attacked by hydroxyl radicals to decolorize the dye molecule. Finally benzene rings and naphthalene rings were mineralized to  $\text{H}_2\text{O}$  and  $\text{CO}_2$  under the oxidation of hydroxyl radicals.

**Key words:** Fenton oxidation; degradation pathway; iron-carbon micro-electrolysis; Direct Pink 12B

### Introduction

Dye wastewater has the typical characteristics of complicated component, high pollutant concentrations, deep chromaticity and poor biodegradability. Therefore, the dye wastewater is the main research objects of industrial wastewater treatment (Wang et al., 2010). The iron-carbon micro-electrolysis (ICME) and Fenton oxidation technology have the characteristics of the low cost, wide applicability and long service life. Moreover, the treatment technology of ICME and Fenton is easy to operate and has important application value (Li et al., 2010; Yu et al., 2011). The main objective of this study was to provide reference for further treatment on the basis of degradation product.

## 1 Experiments

### 1.1 UV-Vis analysis

The ICME reaction was investigated under the following conditions: the simulated wastewater concentration of Direct Pink 12B was 2.0 g/L, the initial pH value was 2, the dosage of scrap iron was 10 g, and the mass ratio of Fe/C was 1:1.1. The reaction was observed at the reaction time of 0 (initial solution), 10, 20, 30, 60 and 120 min, respectively, then the sample was analyzed by UV in the

range of 200 to 800 nm.

The Fenton reaction was studied under the following conditions: the  $\text{H}_2\text{O}_2$  concentration was 37 mmol/L, the pH value was 3, and the dosage of  $\text{FeSO}_4$  was 1.2 mmol. The reaction was investigated at the reaction time of 0 (initial solution), 5, 10, 20 and 30 min, respectively. The 25 mL sample was taken out at the corresponding time point and the  $\text{Na}_2\text{SO}_3$  solution was immediately dropped into the sample to end the reaction. The absorption spectra of the sample solution were scanned by UV in the range of 200 to 800 nm.

### 1.2 Infrared spectroscopic analysis

The ICME experiment was investigated under the following conditions: Direct Pink 12B concentration was 2 g/L, the pH value was 2.0, the dosage of scrap iron was 10 g and the mass ratio of Fe/C was 1:1.1. The samples were taken out at the reaction time of 0 (initial solution) and 120 min respectively. The samples were kilned dry and scanned by Fourier transform infrared spectrometer (WQF-200, Beijing Rayleigh Analytical Instrument Corp. Ltd., China)

The Fenton oxidation experiment was studied under the following conditions: the pH value was 3.0, the concentration of  $\text{H}_2\text{O}_2$  was 37 mmol/L and the dosage of  $\text{FeSO}_4$  was 1.2 mmol. The samples were taken out at the reaction time of 0 (initial solution) and 30 min respectively. The samples were kilned dry and scanned by Fourier transform infrared

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spectrometer.

### 1.3 HPLC-MS analysis

The high performance liquid chromatography (HPLC) analysis meter is Agilent1100 Serial, USA. The degasser type is G1379A. The pressure pump is Quatpump, type G1311A. The detector is DAD, type G1315B. The chromatographic column which including 190–950 nm DAD UV detector is ZORBAX SB-C18 with specification size 4.6 mm × 250 mm, 5 μm. The moving phase is methanol and water with the volume ratio of 60:40. The column temperature is 35°C. The flow velocity is 0.8 mL/min. The injection volume is 5 μL. The detector wavelength is 254 nm.

Mass spectrometry (MS) was operated under the following conditions: electrospray ionization source is Negative ion, scan mode 5 V, the block temperature 200°C, the probe voltage minus 3.5 kV, the detection voltage 1.5 kV, the MS scan area 50–500  $m/z$  and the collision induced dissociation voltage 70 V.

The experiment conditions were the same to the condition of UV-Vis and infrared spectroscopic analysis. The solution sample was taken out at the reaction time of 0–40 min and 0.2 mL of 0.5 mol/L  $\text{Na}_2\text{SO}_3$  solution was immediately dropped into the sample to end the reaction. The absorption spectra of the sample were scanned by HPLC-MS analysis.

## 2 Results

### 2.1 UV-Vis scan analysis

**Figure 1** shows that Direct Pink 12B in the UV-Vis spectral region had five absorption peaks at the wavelength of 212, 226, 302, 390 and 530 nm respectively. The maximum absorption wavelength of the azo bond conjugated group appears at 538 nm. The maximum absorption wavelengths of the benzene ring and the naphthalene ring structures appear at the ultraviolet region, and the presence of naphthalene ring structure causes a red shift of the absorption peak of conjugated double bond. The N=N bond containing  $p$  electrons, the benzene ring and naphthalene ring together form a conjugated chromogen. The characteristic absorption peak of the conjugated chromogen appears at 390 and 530 nm while the characteristic absorption peak of benzene ring appears at 212 nm and the peak of the naphthalene ring at 302 nm.

Azo group is the chromophore of Direct Pink 12B. When the azo group exposed to light, heat and acidic or alkaline medium, it becomes relatively active and unstable. The ground state electrons of azo group are easy to absorb energy and lead to electronic transition. The transition electrons are turned into excited electrons, which make the carbon atoms linked with the azo group become unstable. Therefore, the N–C bonds are disconnected first. That is why the dye molecule is decolorized (Huang et al.,

2001).

The UV-Vis spectrogram of Direct Pink 12B in the ICME treating process for 10 min is shown in **Fig. 1a**. The characteristic absorption peak at the visible region declined obviously while the characteristic absorption peaks at the ultraviolet region declined insignificantly. These indicated that the azo group was destroyed more easily than the benzene ring and naphthalene ring structures. The characteristic absorption peak at 538 nm almost disappeared after being treated by ICME for 120 min. This indicated that the azo bond was destroyed basically. The characteristic absorption peaks value at the region of below 300 nm declined slightly while their shape remained almost unchanged, indicating that ring-opening reaction of the benzene ring and naphthalene ring groups almost did not occur (Liu et al., 2011; Mao et al., 2009; Brillas et al., 2000).

**Figure 1b** shows that the maximum absorption peak of the solution appears at 530 nm before treatment but the peak disappeared after 5 min, indicating that the relatively active azo double bond was first broken in the Fenton oxidation process. As the reaction proceeded, the peak value and shape both changed greatly at the ultraviolet region (212 and 302 nm). This indicated that the benzene ring and naphthalene ring structures were broken rapidly in the Fenton reaction system. The obvious blue shift appeared due to a large number of intermediates such as hydroxide radical ( $\cdot\text{OH}$ ) and polyhydroxy compound.

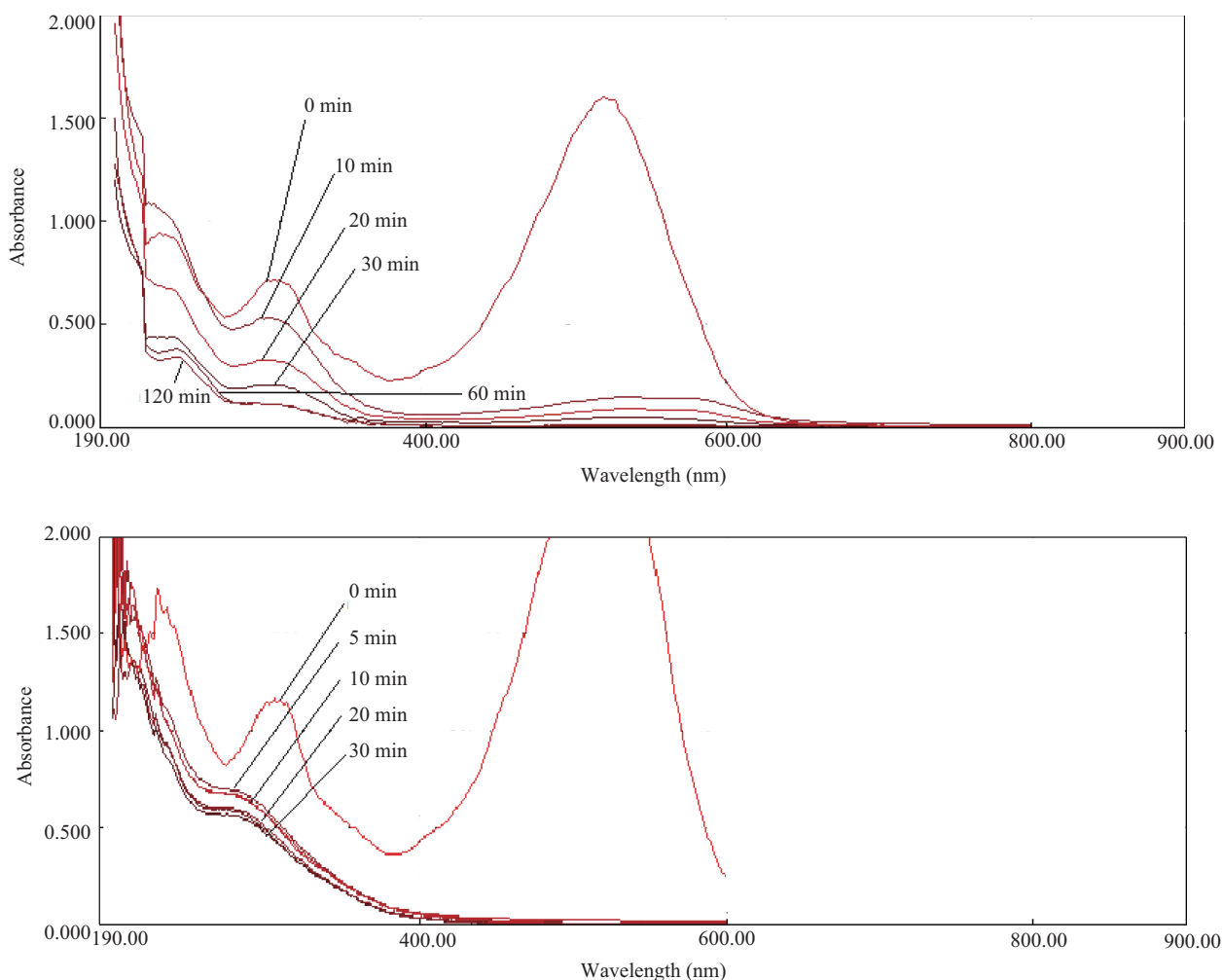
### 2.2 Infrared spectroscopic analysis

The infrared spectrogram of Direct Pink 12B molecular structure is shown in **Fig. 2**. The N=N bonds are the major chromophore of Direct Pink 12B. The characteristic absorption peaks of Direct Pink 12B generally appear at 1379 and 1236  $\text{cm}^{-1}$ . The stretching vibration characteristic absorption peaks of  $-\text{OH}$  generally appear at 3450  $\text{cm}^{-1}$ . The peaks at 1541 and 1489  $\text{cm}^{-1}$ , the major correlative peaks of benzene ring C=C bond stretching vibration, are considered as an expression of benzene ring structure.

**Figure 2a** shows that a strong absorption peak appeared at 1541  $\text{cm}^{-1}$  and thus it can be concluded that the peak was the stretching vibration peak of skeleton of C=C bond. The characteristic absorption peak of C–N linked to benzene ring appeared at 1379  $\text{cm}^{-1}$ ; the peak of 1198  $\text{cm}^{-1}$  was the characteristic absorption peak of 1,3-disubstituted benzene ring. The peaks of 1541  $\text{cm}^{-1}$  and 1489  $\text{cm}^{-1}$  were skeletal vibration absorption peaks of benzene ring and naphthalene ring respectively.

The infrared spectrogram of Direct Pink 12B in the ICME process for 120 min is shown in **Fig. 2b**. The characteristic adsorption peaks of N=N bond at 1379, 1236  $\text{cm}^{-1}$  disappeared but the peak shapes were almost unchanged at other regions. It was concluded that the azo bond had been disconnected, but the benzene ring and naphthalene ring had not been disconnected in the reducing





**Fig. 1** UV-Vis spectra of Direct Pink 12B in ICME process (a) and in Fenton oxidation process (b).

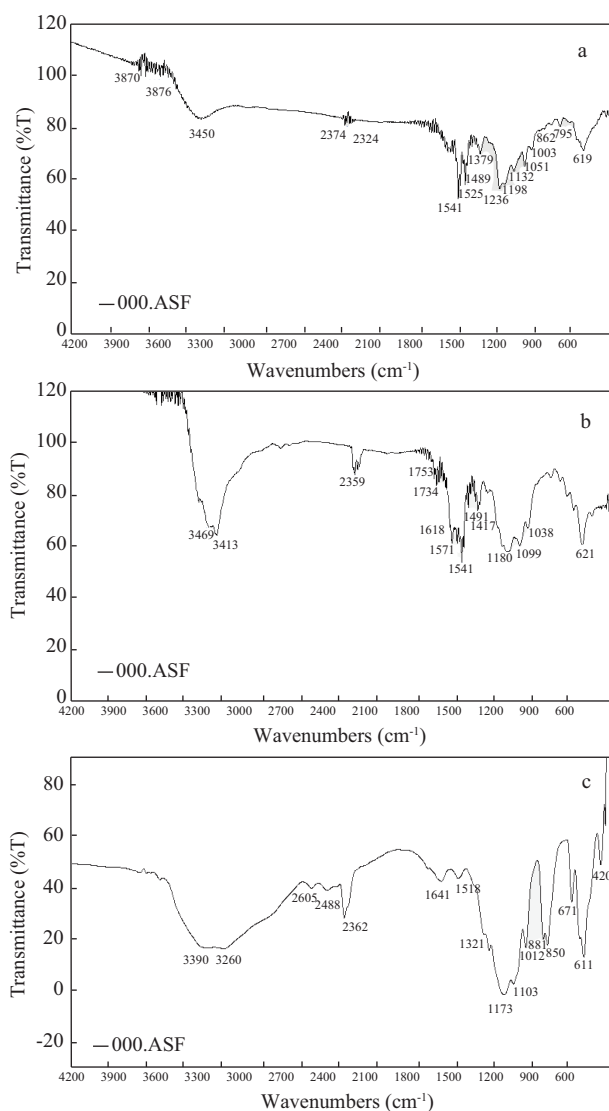
action of ICME.

The infrared spectrogram of Direct Pink 12B in the Fenton oxidation process for 30 min is shown in **Fig. 2c**. The broad adsorption peak at  $3390\text{ cm}^{-1}$  was stretching vibration adsorption peak of  $\cdot\text{OH}$ . The adsorption peaks at  $1417$  and  $860\text{ cm}^{-1}$  were adsorption peaks of  $\text{CO}_3^{2-}$ . Thus it can be concluded that the  $\text{CO}_3^{2-}$  was generated due to the strong oxidation of  $\cdot\text{OH}$  in the Fenton reaction process. A sharp absorption peak and a weak absorption peak were observed at  $1641$  and  $1321\text{ cm}^{-1}$  respectively. It was concluded that the absorption peak at  $1641\text{ cm}^{-1}$  belonged to  $\text{C}=\text{O}$  bond while the peak at  $1321\text{ cm}^{-1}$  belonged to  $\text{C}-\text{O}$  bond. The peaks at  $1670\text{--}1650\text{ cm}^{-1}$  of  $\text{C}=\text{O}$  bond stretching vibration and at  $1320\text{--}1211\text{ cm}^{-1}$  of  $\text{C}-\text{O}$  bond stretching vibration were the expression of  $-\text{COOH}$ . It was concluded that there were some carboxylic intermediates produced in the treatment. The peaks at  $1051$  and  $1186\text{ cm}^{-1}$  becomes smaller, which belong to  $\text{R}-\text{SO}_3$ . The surplus  $\text{BaCl}_2$  was added into the solution for depositing  $\text{SO}_4^{2-}$  to detect the sulfate ion ( $\text{SO}_4^{2-}$ ). When white precipitate emerged, it suggested that  $\text{SO}_4^{2-}$  was indeed generated in the Fenton oxidation process.

### 2.3 HPLC-MS analysis

A typical solution sample had been analyzed with HPLC-MS in the iron-carbon micro-electrolysis and Fenton reaction degradation process. Totally more than 20 intermediate products had been detected and identified by HPLC-MS in **Fig. 3**

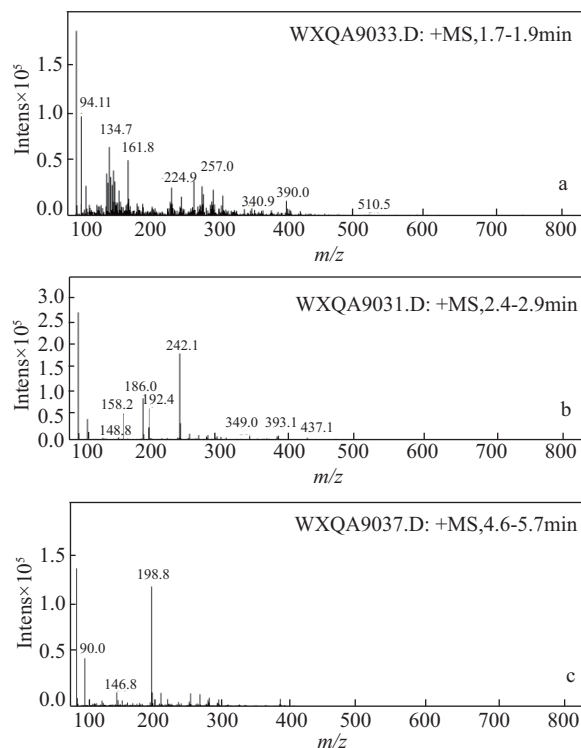
According to **Fig. 3a**, it was concluded that the molecular ion peak of  $m/z$  257.0 belongs to 1,2,6-trihydroxy-3-naphthalene sulfonate ( $\text{C}_{10}\text{H}_7\text{O}_6\text{S}$ ); the molecular ion peak of  $m/z$  94.11 belongs to phenol ( $\text{C}_6\text{H}_6\text{O}$ ). As shown in **Fig. 3b**, the molecular ion peak of  $m/z$  158.2 may belongs to naphthoquinone ( $\text{C}_{10}\text{H}_6\text{O}_2$ ). The naphthoquinone may have two isomers: 1,2-naphthoquinone and 1,4-naphthoquinone. The molecular ion peak of  $m/z$  106 belongs to phenyldiazene, which was the degradation product of Direct Pink 12B with the molecular formula being  $\text{C}_6\text{H}_6\text{N}_2$ . The finding further demonstrated that the  $\text{N}-\text{C}$  bond beside azo bond was first broken by  $\cdot\text{OH}$  in Fenton process. The molecular ion peak of  $m/z$  90 belongs to oxalic acid ( $\text{C}_2\text{H}_2\text{O}_4$ ) shown **Fig. 3c**, and it was the resultant of phenyldiazene and phenol. It was further proven that benzene rings had been degraded into small



**Fig. 2** Infrared spectra of pretreatment Direct Pink 12B in ICME process at 0 min (a), 120 min (b) and the spectra of post treatment Direct Pink 12B in Fenton oxidation process at 30 min (c).

molecule acids.

According to the above HPLC-MS spectrogram, a variety of different intermediate products were produced in Fenton degradation process. The conjugate structure was destroyed in the oxidation reaction process, which led to ring opening of benzene and naphthalene. A number of ion peaks appeared after Direct Pink 12B oxidized by Fenton for 30 min, with the retention time of 1.8–4.7 min through mass spectra. According to the above analysis, it was inferred that the main degradation products were  $C_{10}H_7O_6S$ ,  $C_6H_6O$ ,  $C_{10}H_6O_2$ ,  $C_6H_6N_2$  and  $C_2H_2O_4$ , which is in line with the analysis results by the UV, IR, HPLC spectrogram.



**Fig. 3** HPLC-MS spectra of Direct Pink 12B with the retention time at 1.8 (a), 2.6 (b), and 4.7 min (c).

### 3 Discussion

The degradation pathways can be proposed based on the dye molecular structure and electrophilic theory according to the experimental results by UV-Vis, IR, HPLC-MS. (1) The N–C bonds connected with azo group beside naphthalene rings were first attacked by  $\cdot OH$  and broken, thus the dye molecular cut into phenyldiazene and a part that remains. (2) The phenyldiazene was very unstable and easily oxidized by  $\cdot OH$  into phenol and  $N_2$ . Phenol was further attacked by  $\cdot OH$  to be converted into hydroquinone and pyrocatechol. Then the hydroquinone and pyrocatechol were oxidized by  $\cdot OH$  into oxalic acid. Finally oxalic acid was mineralized into inorganic ion. (3) In the oxidation process,  $-NH-$  was finally mineralized into  $NO_3^-$ .  $-SO_3^-$  connected with naphthalene ring was attacked by  $\cdot OH$ , and was replaced by  $\cdot OH$  to generate the intermediate products (polyhydroxy naphthalene sulfonate). The intermediate was attacked by  $\cdot OH$  to generate the isomerides 1,4-naphthoquinone and 1,2-naphthoquinone. The naphthoquinone was very unstable in the oxidation system and easily oxidized by  $\cdot OH$  into small molecules, such as salicylic acid, phthalaldehyde, several small esters and oxalic acid. Finally these small molecules were mineralized into inorganic ion, too. The degradation pathway is shown in Fig. 4.

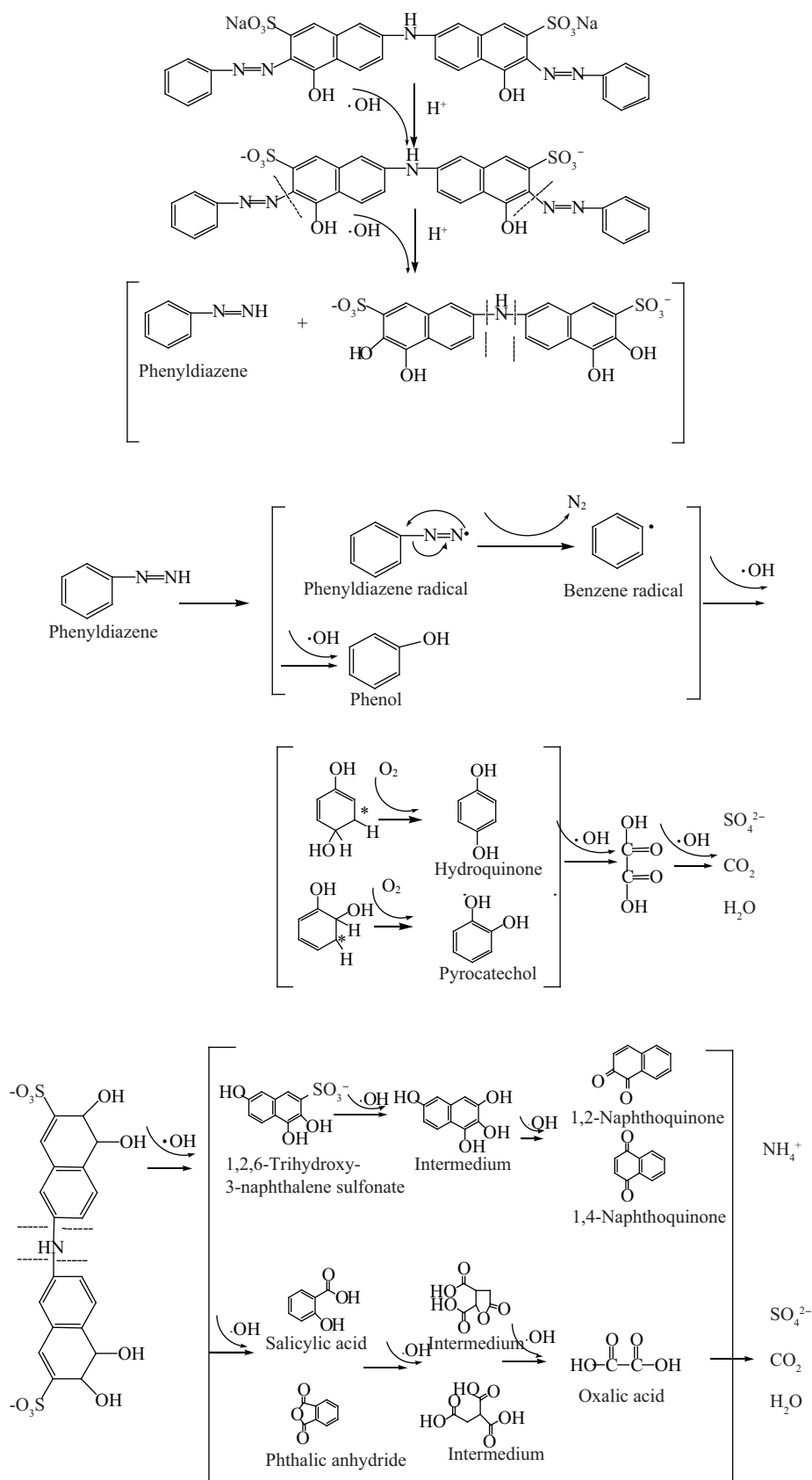


Fig. 4 Degradation process of Direct Pink 12B.



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