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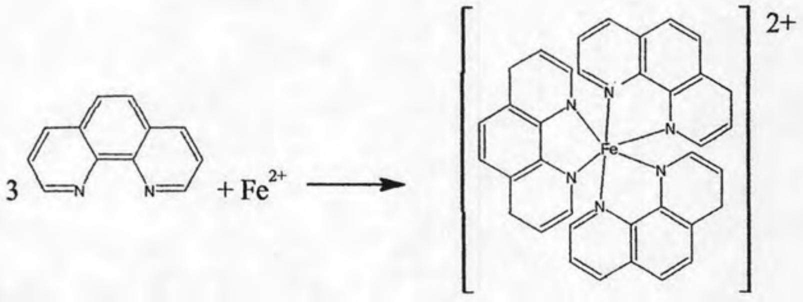
APPENDICES

APPENDIX A

Analysis of ferrous ions (APHA, 1992)

Principle

The phenanthroline method is the preferred standard procedure for the measurement of iron in water at the present time, except when color, turbidity, phosphate, or heavy metal interferences are present. The method depends upon the fact that 1,10-phenanthroline combine with ferrous to form a complex iron that is orange-red in color. Three molecules of 1,10-phenanthroline are required to sequester or form a complex ion with each ferrous ion. The reaction may be represented as shown in the following Equation:



(A-1)

Reagents

1. Hydrochloric acid, HCl: 20 ml of concentration hydrochloric acid was diluted to 1,000 ml with RO water.
2. Ammonium acetate buffer solution: 500 g of $NH_4C_2H_3O_2$ was dissolved in 300 ml of RO water. Then 1,400 ml of concentration acetic acid was filled up to the mark of 2,000 ml.
3. Phenanthroline solution: dissolve 5 g of 1,10-phenanthroline hydrochloride ($C_{12}H_8N_2$•$HCl+H_2O$) into 1,000 ml of RO water.

Apparatus

1. Spectrophotometer (Shimadsu UV-1201) The absorbance used for ferrous ion analysis was 510 nm.
2. 50 ml Erlenmeyer flask
3. Medicine dropper

Procedure

1. 25 ml of HCl from stock solution was prepared in 50 ml volumetric flask. Then, 10 ml of phenanthroline solution and 5 ml of ammonium acetate solution were added with vigorous stirring.
2. 1 ml of sample was filled and diluted to 50 ml with RO water. After that, it had to stand for 10 min. do not expose to sunlight. (Color develop was rapid in the presence of excess phenanthrolines.)
3. Measured samples by spectrophotometer at 510 nm.

Preparing for Calibration Curve

The standard ferrous solution were prepared in the range of 0 to 5 mg/l as Fe^{2+}

1. 25 ml of HCl from stock solution was prepared in five 50 ml volumetric flasks. Then, 10 ml of phenanthroline solution and 5 ml of ammonium acetate solution were added with vigorous stirring.
2. 1 ml of 0, 0.5, 1, 3, and 5 mg/l as Fe^{2+} standard solution were pipetted, respectively.
3. The samples were diluted to 50 ml with RO water, mixed thoroughly. After that, it had to stand for 10 min.
4. Measured samples by spectrophotometer at 510 nm.

APPENDIX B

Standard Iodometric (Kingzett CT, 1880 and Kolthoff IM, 1920)

Principle

Hydrogen peroxide oxidizes iodine in the presence of acid and molybdate catalyst. The iodine formed is titrated with thiosulfate solution, incorporating a starch indicator as demonstrated in the following Equation:

$$H_2O_2 + 2KI + Na_2SO_4 \longrightarrow I_2 + K_2SO_4 + 2H_2O \qquad \text{(B-1)}$$

$$I_2 + 2\,Na_2S_2O_3 \longrightarrow Na_2S_4O_6 + 2NaI \qquad \text{(B-2)}$$

Interferences

Other oxidizing agents will also produce iodine, whereas reducing agents (and unsaturated organics) will react with the liberated iodine. The contribution from other oxidizing agents can be determinated by containing the acid and molybdate catalyst.

Reagents

1. Potassium iodide solution (1% w/v): dissolve 10 gram KI into 1 L of RO water.
2. Ammonium molybdate solution: dissolve 9 grams ammonium molybdate in 10 ml 6N NH_4OH, add 24 grams NH_4NO_3 and dilute to 100 ml with RO water.
3. Sulfuric acid solution (1:4 H_2SO_4): carefully add one part H_2SO_4 98% to four parts RO water.
4. Starch indicator
5. Sodium thiosulfate solution (0.0125 N)

Apparatus

1. Analytical balance (+/- mg/l)
2. Small weighing bottle (< 5 ml)
3. 250 ml Erlenmeyer flask
4. 50 ml burette (Class A)
5. Medicine dropper

Procedure

1. Transfer sample to Erlenmeyer flask.
2. Add to Erlenmeyer flask 50 ml of RO water. Next, 10 ml of sulfuric acid solution and 15 ml of potassium iodide were added. Then two drops ammonium molybdate solution was added.
3. Titrate with 0.0125 N sodium thiosulfate to faint yellow or straw color. Swirl or stir gently during titration to minimize iodine loss.
4. Add about 2 ml starch indicator, and continue titration until the blue color just disappears.
5. Repeat steps 2-4 on a blank sample of water.
6. Note ml of 0.0125 N $Na_2S_2O_3$ for samples and blanks analysis.

Calculation

$$H_2O_2,\ mg/l = \frac{(A-B) \times N \times 17 \times 1000}{ml.\ sample}$$

where:

A = ml of $Na_2S_2O_3$ for sample

B = ml of $Na_2S_2O_3$ for blank

N = Normality of $Na_2S_2O_3$

APPENDIX C

Chemical Oxygen Demand (COD) ANALYSIS

(Closed-reflux titrimetric method based on the Standard methods, APHA, 1992).

Principle

The chemical oxygen demand (COD) test is commonly used to indirectly measure the amount of organic compounds in water. Most applications of COD determine the amount of organic pollutants found in surface water (e.g. lakes and rivers), making COD a useful measure of water quality. It is expressed in milligrams per liter (mg/L), which indicates the mass of oxygen consumed per liter of solution. Older references may express the units as parts per million (ppm). The basis for the COD test is that nearly all organic compounds can be fully oxidized to carbon dioxide with a strong oxidizing agent under acidic conditions.

For many years, the strong oxidizing agent potassium permanganate ($KMnO_4$) was used for measuring chemical oxygen demand. Measurements were called oxygen consumed from permanganate, rather than the oxygen demand of organic substances. Potassium permanaganate's effectiveness at oxidizing organic compounds varied widely, and in many cases biochemical oxygen demand (BOD) measurements were often much greater than results from COD measurements. This indicated that potassium permanganate was not able to effectively oxidize all organic compounds in water, rendering it a relatively poor oxidizing agent for determining COD. Since then, other oxidizing agents such as ceric sulfate, potassium iodate, and potassium dichromate have been used to determine COD. Of these, potassium dichromate ($K_2Cr_2O_7$) has been shown to be the most effective: it is relatively cheap, easy to purify, and is able to nearly completely oxidize almost all organic compounds.

For all organic matter to be completely oxidized, an excess amount of potassium dichromate (or any oxidizing agent) must be present. Once oxidation is complete, the amount of excess potassium dichromate must be measured to ensure that the amount of Cr^{3+} can be determined with accuracy. To do so, the excess potassium dichromate is titrated with ferrous ammonium sulfate (FAS) until all of the excess oxidizing agent has been reduced to Cr^{3+}.

Typically, the oxidation-reduction indicator Ferroin is added during this titration step as well. Once all the excess dichromate has been reduced, the Ferroin indicator changes from blue-green to reddish-brown. The amount of ferrous ammonium sulfate added is equivalent to the amount of excess potassium dichromate added to the original sample

Reagent

The reagent uses in COD measurement are:

1. $K_2Cr_2O_7$ (0.1 N): 167 ml H_2SO_4 add into 1000 ml volumetric flask and then add 200 ml of DI water. After that 4.913 gram of $K_2Cr_2O_7$ was added and then 33.3 ml of $HgSO_4$. Then DI water was adjusted up-to 1 liter.
2. H_2SO_4 solution: prepared by added 25.3 gram of Ag_2SO_4 into 2.5 liter of H_2SO_4
3. FAS (0.025 N): 9.8 gram of $FeSO_4(NH_4)SO_4.6H_2O$ was put into 1 liter volumetric flask and then 20 ml of H_2SO_4 was added. Then the DI water was poured until 1 liter.
4. Ferroin Indicator: 1 drop

Procedure

1. The sample 2.5 ml was put into tube
2. Add 1.5 ml of $K_2Cr_2O_7$ (0.1 N), 3.5 ml of H_2SO_4 reagent were
3. Incubate the mixed chemical at 150°C for 2 hours
4. Then the chemical was put outside at room temperature to cool down the temperature
5. After that, 1 drop of Ferroin indicator was added and titrated by using FAS.

Calculation

The following formula is used to calculate COD:

$$COD = \frac{(blank - sample) * FAS\ conc. * 8000}{2.5}$$

$$FAS\ conc. = \frac{0.1 * 1.5}{V\ FAS\ (normal)}$$

where blank is the volume of FAS used in the blank sample, *sample* is the volume of FAS in the original sample, and *FAS conc.* is the normality of FAS. If milliliters are used consistently for volume measurements, the result of the COD calculation is given in mg/L.

APPENDIX D

(D-1) Carrier comparison

Table D-1 The carrier comparison onto the 2,6-dimethylanline oxidation

Time	2,6-DMA removal, C/C_0						
min	Fenton	Al_2O_3	SiO_2	Black	White	Brown	Colour
0	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
2	0.5054	0.5312	0.6032	0.6767	0.5166	0.3706	0.3182
5	0.4797	0.4743	0.5974	0.2787	0.4850	0.3283	0.3064
10	0.4750	0.4203	0.5960	0.2475	0.3079	0.2488	0.2994
20	0.4639	0.3814	0.4672	0.1946	0.3057	0.2196	0.2895
40	0.4523	0.3585	0.4069	0.1623	0.2812	0.2121	0.1901
60	0.4174	0.2750	0.3741	0.1456	0.2575	0.1501	0.1108
90	0.3480	0.2527	0.3344	0.1370	0.1564	0.0832	0.0551
120	0.3175	0.2442	0.2895	0.1312	0.1253	0.0598	0.0021
150	0.2951	0.1599	0.2831	0.1085	0.0964	0.0338	0.0000
180	0.2919	0.1229	0.1794	0.0641	0.0806	0.0225	0.0000
240	0.2196	0.0491	0.1612	0.0600	0.0334	0.0000	0.0000
300	0.1973	0.0087	0.1532	0.0410	0.0043	0.0000	0.0000
%Efficiency	80.2748	99.1268	84.6793	95.9049	99.5697	100.0000	100.0000

Note : [2,6-DMA] = 1 mM, $[Fe^{2+}]$ = 1 mM, $[H_2O_2]$ = 20 mM, pH = 2 and Al_2O_3 = 74.07 g/l

Table D-2 The carrier comparison onto the total iron removal

Time	Total iron removal, C/C_0						
min	Fenton	Al_2O_3	SiO_2	Black	White	Brown	Colour
0	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
2	1.0000	0.9716	0.9141	0.7912	0.7460	0.9206	0.8157
5	0.9848	0.9672	0.8616	0.7885	0.7640	1.0337	0.8350
10	0.9682	0.9596	0.8076	0.7995	0.7924	0.9370	0.7787
20	0.9671	0.9117	0.7919	0.8290	0.7465	0.9613	0.8603
40	0.9656	0.8640	0.7360	0.7786	0.7841	0.8798	0.8217
60	0.9537	0.8441	0.7032	0.8812	0.7181	0.8804	1.0151
90	0.9532	0.8223	0.6923	0.9172	0.7485	1.0114	0.8504
120	0.9457	0.7785	0.6697	0.8271	0.7044	0.9773	0.9029
150	0.9409	0.7545	0.6651	0.8363	0.6557	1.0028	1.1043
180	0.9328	0.7429	0.6565	0.8267	0.7591	1.0221	0.9997
240	0.9030	0.6809	0.6559	0.8022	0.7143	0.9833	1.2077
300	0.8540	0.6338	0.5915	0.7830	0.7547	1.0371	1.2956
%Efficiency	14.6020	36.6197	40.8532	21.7000	24.5350	ND	ND

Note : [2,6-DMA] = 1 mM, $[Fe^{2+}]$ = 1 mM, $[H_2O_2]$ = 20 mM, pH = 2 and Al_2O_3 = 74.07 g/l

Table D-3 The carrier comparison onto the ferrous ion removal

Time	Ferrous ion removal, C/C_0						
min	Fenton	Al_2O_3	SiO_2	Black	White	Brown	Colour
0	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
2	0.9979	0.9619	0.8739	0.9675	0.8656	1.1033	0.6824
5	0.9934	0.9481	0.8273	0.5296	0.6596	0.9915	0.6939
10	0.9927	0.9272	0.8127	0.3068	0.6175	0.9030	0.8451
20	0.9904	0.8823	0.7503	0.2381	0.6132	0.9551	0.8487
40	0.9904	0.8706	0.7366	0.3570	0.5763	0.8950	0.7947
60	0.9789	0.8241	0.7331	0.1167	0.5948	0.9180	0.7819
90	0.9716	0.8118	0.7251	0.1870	0.5449	0.8702	0.7144
120	0.9682	0.7765	0.7172	0.0729	0.5610	1.0253	0.7578
150	0.9657	0.7646	0.6926	0.0762	0.3760	0.9563	0.7854
180	0.9657	0.7462	0.6828	0.0649	0.4903	0.9474	0.9156
240	0.9637	0.6730	0.6563	0.0504	0.2478	1.0606	0.7304
300	0.9328	0.5710	0.5304	0.1600	0.2244	0.9453	0.8291
%Efficiency	6.7206	42.8952	46.9591	84.0000	77.5640	5.4700	17.0930

Note : [2,6-DMA] = 1 mM, $[Fe^{2+}]$ = 1 mM, $[H_2O_2]$ = 20 mM, pH = 2 and Al_2O_3 = 74.07 g/l

Table D-4 The carrier comparison onto the hydrogen peroxide depletion

Time	H_2O_2 depletion, C/C_0						
min	Fenton	Al_2O_3	SiO_2	Black	White	Brown	Colour
0	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
2	0.9971	0.9724	0.9878	0.9643	0.9563	0.9960	0.9975
5	0.9806	0.9642	0.9878	0.9286	0.9027	0.9919	0.9357
10	0.9765	0.9518	0.9838	0.9152	0.8986	1.0000	0.9275
20	0.9683	0.9353	0.9680	0.8929	0.9027	0.9919	0.8945
40	0.9683	0.8900	0.9680	0.8750	0.8986	0.9960	0.8409
60	0.9642	0.8735	0.9640	0.8527	0.8945	0.9879	0.8244
90	0.9559	0.8694	0.9364	0.8170	0.8821	0.9798	0.8162
120	0.9312	0.8653	0.9087	0.7679	0.8739	0.9556	0.8120
150	0.8982	0.8570	0.8613	0.7589	0.8656	0.9274	0.7643
180	0.8817	0.7829	0.8495	0.7545	0.8739	0.9113	0.7439
240	0.8735	0.6634	0.8455	0.7366	0.8533	0.8226	0.6844
300	0.8405	0.6222	0.8139	0.7277	0.8409	0.8065	0.6348
%Efficiency	15.9456	37.7833	18.6092	27.2321	15.9110	19.3548	36.5210

Note : [2,6-DMA] = 1 mM, $[Fe^{2+}]$ = 1 mM, $[H_2O_2]$ = 20 mM, pH = 2 and Al_2O_3 = 74.07 g/l

(D-2) Control experiment

Table D-5 Control experiment of 2,6-dimethylaniline in fluidized-bed Fenton

Time	2,6-DMA removal, C/C_0			
min	Fe^{2+} 0 mM	H_2O_2 0 mM	Fenton	Adsorption
0	1.0000	1.0000	1.0000	1.0000
2	0.9750	0.9770	0.5050	0.9900
5	0.9540	0.9670	0.4800	0.9870
10	0.9420	0.9460	0.4750	0.9780
20	0.9310	0.9140	0.4640	0.9730
40	0.8760	0.8830	0.4520	0.9660
60	0.8740	0.8650	0.4170	0.9610
90	0.8120	0.8120	0.3480	0.9590
120	0.8080	0.8060	0.3180	0.9520
150	0.7810	0.7960	0.2950	0.9480
%Efficiency	21.8520	20.4372	70.4945	5.1574

Note : [2,6-DMA] = 1 mM, $[Fe^{2+}]$ = 5 mM, $[H_2O_2]$ = 15 mM, pH = 3 and Al_2O_3 = 74.07 g/l

(D-3) Effect of pH

Table D-6 The effects of pH on the 2,6-dimethylaniline oxidation in fluidized-bed reactor; 2,6-Dimethylaniline removal

Time	2,6-DMA removal, C/C_0			
min	pH 1	pH 2	pH 3	pH 4
0	1.0000	1.0000	1.0000	1.0000
2	0.7247	0.0582	0.0166	0.5490
5	0.1739	0.0396	0.0075	0.0289
10	0.1730	0.0225	0.0000	0.0155
20	0.1424	0.0000	0.0000	0.0155
40	0.1422	0.0000	0.0000	0.0104
60	0.1330	0.0000	0.0000	0.0055
90	0.1188	0.0000	0.0000	0.0053
120	0.1149	0.0000	0.0000	0.0000
150	0.0984	0.0000	0.0000	0.0000
%Efficiency	90.1581	97.7541	99.2539	99.4733

Note : [2,6-DMA] = 1 mM, $[Fe^{2+}]$ = 5 mM, $[H_2O_2]$ = 15 mM, pH = 3 and Al_2O_3 = 74.07 g/l

Table D-7 The effects of pH on the 2,6-dimethylaniline oxidation in fluidized-bed reactor; Total iron removal

Time	Total iron removal, C/C_0			
min	pH 1	pH 2	pH 3	pH 4
0	1.0000	1.0000	1.0000	1.0000
2	0.9756	0.9753	0.9508	0.9321
5	0.9861	0.9491	0.9309	0.9157
10	0.9761	0.9372	0.9051	0.8916
20	0.9622	0.8968	0.8802	0.8178
40	0.9294	0.9158	0.8166	0.9057
60	0.9615	0.9450	0.7441	0.8534
90	0.9577	0.9024	0.7170	0.8155
120	0.9455	0.8867	0.7166	0.8695
150	0.9129	0.8610	0.7160	0.8703
%Efficiency	8.7145	13.8973	28.4000	12.9744

Note : [2,6-DMA] = 1 mM, $[Fe^{2+}]$ = 5 mM, $[H_2O_2]$ = 15 mM, pH = 3 and Al_2O_3 = 74.07 g/l

Table D-8 The effects of pH on the 2,6-dimethylaniline oxidation in fluidized-bed reactor; Ferrous ion removal

Time	Ferrous ion removal, C/C_0			
min	pH 1	pH 2	pH 3	pH 4
0	1.0000	1.0000	1.0000	1.0000
2	0.9908	0.9761	0.8990	0.3252
5	0.9836	0.9694	0.7678	-0.0050
10	0.9842	0.9743	0.6673	0.0114
20	0.9760	0.9620	0.5594	-0.0105
40	0.9316	0.9360	0.3872	-0.0111
60	0.9677	0.9006	0.2148	-0.0152
90	0.9625	0.9147	0.1491	-0.0166
120	0.9432	0.8881	0.1285	-0.0226
150	0.9273	0.8734	0.1280	-0.0193
%Efficiency	7.2681	12.6612	87.2000	ND

Note : [2,6-DMA] = 1 mM, $[Fe^{2+}]$ = 5 mM, $[H_2O_2]$ = 15 mM, pH = 3 and Al_2O_3 = 74.07 g/l

Table D-9 The effects of pH on the 2,6-dimethylaniline oxidation in fluidized-bed reactor; Hydrogen peroxide depletion

Time	H_2O_2 depletion, C/C_0			
min	pH 1	pH 2	pH 3	pH 4
0	1.0000	1.0000	1.0000	1.0000
2	0.3175	0.7769	0.2895	0.2755
5	0.2924	0.7321	0.2295	0.2382
10	0.2883	0.7149	0.2272	0.2312
20	0.2866	0.6632	0.1177	0.2464
40	0.2831	0.3928	0.0641	0.2621
60	0.2924	0.1585	0.0559	0.2569
90	0.2895	0.0000	0.0326	0.2289
120	0.2971	0.0000	0.0128	0.2470
150	0.2912	0.0000	0.0128	0.2167
%Efficiency	70.8760	100.0000	98.7185	78.3318

Note : [2,6-DMA] = 1 mM, $[Fe^{2+}]$ = 5 mM, $[H_2O_2]$ = 15 mM, pH = 3 and Al_2O_3 = 74.07 g/l

(D-4) Effect of initial ferrous ion concentration

Table D-10 The effects of initial ferrous ion concentration on the 2,6-dimethylaniline oxidation in fluidized-bed reactor; 2,6-Dimethylaniline removal

Time	2,6-DMA removal, C/C_0		
min	Fe^{2+} 1 mM	Fe^{2+} 2.5 mM	Fe^{2+} 5 mM
0	1.0000	1.0000	1.0000
2	0.3691	0.2143	0.0166
5	0.3335	0.1815	0.0075
10	0.3137	0.1464	0.0000
20	0.2832	0.1217	0.0000
40	0.2614	0.1064	0.0000
60	0.1854	0.0846	0.0000
90	0.1826	0.0845	0.0000
120	0.1810	0.0471	0.0000
150	0.1717	0.0456	0.0000
%Efficiency	0.8283	0.9545	1.0000

Note : [2,6-DMA] = 1 mM, $[H_2O_2]$ = 15 mM, pH = 3 and Al_2O_3 = 74.07 g/l

Table D-11 The effects of initial ferrous ion concentration on the 2,6-dimethylaniline oxidation in fluidized-bed reactor; COD removal

Time	COD, C/C_0		
min	Fe^{2+} 1 mM	Fe^{2+} 2.5 mM	Fe^{2+} 5 mM
0	1.0000	1.0000	1.0000
2	0.8636	0.6842	0.8583
5	0.8636	0.6053	0.7165
10	0.8182	0.5263	0.7165
20	0.7727	0.4737	0.7008
40	0.6818	0.4737	0.6535
60	0.5909	0.4737	0.6535
90	0.4545	0.4211	0.6220
120	0.2727	0.2368	0.5118
150	0.2727	0.2105	0.3858
%Efficiency	72.7273	78.9474	61.4173

Note : [2,6-DMA] = 1 mM, $[H_2O_2]$ = 15 mM, pH = 3 and Al_2O_3 = 74.07 g/l

Table D-12 The effects of initial ferrous ion concentration on the 2,6-dimethylaniline oxidation in fluidized-bed reactor; Total iron removal

Time	Total iron removal, C/C_0		
min	Fe^{2+} 1 mM	Fe^{2+} 2.5 mM	Fe^{2+} 5 mM
0	1.0000	1.0000	1.0000
2	0.8420	0.8050	0.9120
5	0.7460	0.8480	0.8970
10	0.6740	0.7770	0.8530
20	0.6190	0.7000	0.8110
40	0.5000	0.6810	0.7570
60	0.3910	0.6750	0.7390
90	0.3220	0.6680	0.7120
120	0.2950	0.6580	0.7220
150	0.3560	0.6540	0.7200
%Efficiency	64.4080	34.6280	27.9610

Note : [2,6-DMA] = 1 mM, $[H_2O_2]$ = 15 mM, pH = 3 and Al_2O_3 = 74.07 g/l

Table D-13 The effects of initial ferrous ion concentration on the 2,6-dimethylaniline oxidation in fluidized-bed reactor; Ferrous ion removal

Time	Ferrous ion removal, C/C_0		
min	Fe^{2+} 1 mM	Fe^{2+} 2.5 mM	Fe^{2+} 5 mM
0	1.0000	1.0000	1.0000
2	0.8690	0.6740	0.8590
5	0.7690	0.4560	0.6860
10	0.6360	0.2860	0.5020
20	0.5220	0.1190	0.3770
40	0.3690	0.0860	0.2190
60	0.2230	0.0690	0.1650
90	0.0870	0.0450	0.1470
120	0.0340	0.0500	0.1330
150	0.0510	0.0410	0.1170
%Efficiency	94.9350	95.9330	88.3170

Note : [2,6-DMA] = 1 mM, $[H_2O_2]$ = 15 mM, pH = 3 and Al_2O_3 = 74.07 g/l

Table D-14 The effects of initial ferrous ion concentration on the 2,6-dimethylaniline oxidation in fluidized-bed reactor; Hydrogen peroxide depletion

Time	H_2O_2 depletion, C/C_0		
min	Fe^{2+} 1 mM	Fe^{2+} 2.5 mM	Fe^{2+} 5 mM
0	1.0000	1.0000	1.0000
2	0.7862	0.7716	0.0000
5	0.7586	0.5744	0.0000
10	0.7000	0.5433	0.0000
20	0.6828	0.5260	0.0000
40	0.6793	0.4810	0.0000
60	0.6517	0.3772	0.0000
90	0.6241	0.3737	0.0000
120	0.5345	0.3114	0.0000
150	0.4552	0.2457	0.0000
%Efficiency	54.4828	75.4325	100.0000

Note : [2,6-DMA] = 1 mM, $[H_2O_2]$ = 15 mM, pH = 3 and Al_2O_3 = 74.07 g/l

(D-5) Effects of initial hydrogen peroxide concentration

Table D-15 The effects of initial hydrogen peroxide concentration on the 2,6-dimethylaniline oxidation in fluidized-bed reactor; 2,6-Dimethylaniline removal

Time	2,6-DMA removal, C/C_0					
min	H_2O_2 1mM	H_2O_2 2.5mM	H_2O_2 5mM	H_2O_2 10mM	H_2O_2 15mM	H_2O_2 20mM
0	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
2	0.8572	0.3583	0.1239	0.1316	0.0490	0.0000
5	0.7556	0.3561	0.1211	0.0994	0.0168	0.0000
10	0.7078	0.3442	0.1189	0.0426	0.0000	0.0000
20	0.7048	0.3395	0.1171	0.0155	0.0000	0.0000
40	0.6877	0.3354	0.1155	0.0000	0.0000	0.0000
60	0.6748	0.3221	0.0955	0.0000	0.0000	0.0000
90	0.6633	0.3100	0.0949	0.0000	0.0000	0.0000
120	0.6420	0.2904	0.0924	0.0000	0.0000	0.0000
150	0.6058	0.2667	0.0710	0.0000	0.0000	0.0000
%Efficiency	39.4160	73.3315	92.8992	100.0000	100.0000	100.0000

Note : [2,6-DMA] = 1 mM, $[Fe^{2+}]$ = 5 mM, pH = 3 and Al_2O_3 = 74.07 g/l

Table D-16 The effects of initial hydrogen peroxide concentration on the 2,6-dimethylaniline oxidation in fluidized-bed reactor; COD removal

Time	COD removal, C/C_0					
min	H_2O_2 1mM	H_2O_2 2.5mM	H_2O_2 5mM	H_2O_2 10mM	H_2O_2 15mM	H_2O_2 20mM
0	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
2	1.0000	0.7412	0.8583	0.7572	0.8763	0.8901
5	0.8795	0.7412	0.7165	0.7457	0.8144	0.8242
10	0.8554	0.7388	0.7165	0.6185	0.7320	0.5165
20	0.8313	0.6706	0.7008	0.5954	0.7320	0.4945
40	0.7831	0.6235	0.6535	0.5954	0.6907	0.4725
60	0.7831	0.6000	0.5906	0.4451	0.5670	0.4505
90	0.7831	0.5529	0.5433	0.3988	0.4845	0.3626
120	0.7590	0.5294	0.4803	0.3873	0.4639	0.3407
150	0.7349	0.5059	0.4173	0.3642	0.4433	0.2967
%Efficiency	26.5060	49.4118	58.2677	63.5838	55.6701	70.3297

Note : [2,6-DMA] = 1 mM, $[Fe^{2+}]$ = 5 mM, pH = 3 and Al_2O_3 = 74.07 g/l

Table D-17 The effects of initial hydrogen peroxide concentration on the 2,6-dimethylaniline oxidation in fluidized-bed reactor; Total iron removal

Time	Total iron removal, C/C_0					
min	H_2O_2 1mM	H_2O_2 2.5mM	H_2O_2 5mM	H_2O_2 10mM	H_2O_2 15mM	H_2O_2 20mM
0	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
2	0.9606	0.9812	0.9123	0.8967	0.9266	0.9296
5	0.9280	0.9020	0.8974	0.8815	0.8920	0.8781
10	0.9006	0.8579	0.8527	0.8216	0.8316	0.8670
20	0.9058	0.8228	0.8108	0.7655	0.8010	0.8285
40	0.8408	0.7595	0.7569	0.6999	0.7839	0.7602
60	0.7957	0.7450	0.7386	0.6985	0.7611	0.7434
90	0.7785	0.7212	0.7216	0.6979	0.7603	0.7195
120	0.7526	0.7210	0.7204	0.6964	0.7459	0.6955
150	0.7168	0.6791	0.7117	0.6934	0.7391	0.6721
%Efficiency	28.3246	32.0896	28.8295	30.6630	26.0886	32.7897

Note : [2,6-DMA] = 1 mM, $[Fe^{2+}]$ = 5 mM, pH = 3 and Al_2O_3 = 74.07 g/l

Table D-18 The effects of initial hydrogen peroxide concentration on the 2,6-dimethylaniline oxidation in fluidized-bed reactor; Ferrous ion removal

Time	Ferrous ion removal, C/C_0					
min	H_2O_2 1mM	H_2O_2 2.5mM	H_2O_2 5mM	H_2O_2 10mM	H_2O_2 15mM	H_2O_2 20mM
0	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
2	0.8856	0.9227	0.8586	0.8440	0.7775	0.8267
5	0.8623	0.7473	0.6856	0.6801	0.5801	0.6532
10	0.7960	0.6244	0.5021	0.5053	0.4414	0.5523
20	0.7687	0.5380	0.3768	0.3225	0.2678	0.4225
40	0.7115	0.4207	0.2194	0.2058	0.1469	0.2748
60	0.6785	0.4114	0.1649	0.1455	0.1300	0.2243
90	0.6114	0.3750	0.1472	0.1355	0.0980	0.1909
120	0.6073	0.3637	0.1328	0.1280	0.0842	0.1545
150	0.5890	0.3345	0.1168	0.1213	0.0718	0.1295
%Efficiency	41.0993	66.5531	88.3172	87.8728	92.8151	87.0519

Note : [2,6-DMA] = 1 mM, $[Fe^{2+}]$ = 5 mM, pH = 3 and Al_2O_3 = 74.07 g/l

Table D-19 The effects of initial hydrogen peroxide concentration on the 2,6-dimethylaniline oxidation in fluidized-bed reactor; Hydrogen peroxide depletion

Time	H_2O_2 depletion, C/C_0					
min	H_2O_2 1mM	H_2O_2 2.5mM	H_2O_2 5mM	H_2O_2 10mM	H_2O_2 15mM	H_2O_2 20mM
0	1.0000	1.0000	1.0000	1.0000	1.0000	1.0000
2	0.0000	0.0000	0.0000	0.8708	0.8791	0.8773
5	0.0000	0.0000	0.0000	0.8465	0.6478	0.7186
10	0.0000	0.0000	0.0000	0.7875	0.5393	0.5887
20	0.0000	0.0000	0.0000	0.7320	0.4338	0.2641
40	0.0000	0.0000	0.0000	0.5828	0.1403	0.0938
60	0.0000	0.0000	0.0000	0.3400	0.0477	0.0476
90	0.0000	0.0000	0.0000	0.1145	0.0347	0.0404
120	0.0000	0.0000	0.0000	0.0312	0.0000	0.0404
150	0.0000	0.0000	0.0000	0.0000	0.0000	0.0000
%Efficiency	100.0000	100.0000	100.0000	100.0000	100.0000	100.0000

Note : [2,6-DMA] = 1 mM, $[Fe^{2+}]$ = 5 mM, pH = 3 and Al_2O_3 = 74.07 g/l

(D-6) Effects of 2,6-dimethylaniline concentration

Table D-15 The effects of initial hydrogen peroxide concentration on the 2,6-dimethylaniline oxidation in fluidized-bed reactor; 2,6-Dimethylaniline removal

Time	2,6-DMA removal, C/C_0		
min	2,6-DMA 0.5 mM	2,6-DMA 1 mM	2,6-DMA 5 mM
0	1.0000	1.0000	1.0000
2	0.0933	0.0861	0.5596
5	0.0224	0.0842	0.5354
10	0.0000	0.0826	0.5346
20	0.0000	0.0814	0.4591
40	0.0000	0.0802	0.4493
60	0.0000	0.0663	0.4336
90	0.0000	0.0659	0.4244
120	0.0000	0.0642	0.4154
150	0.0000	0.0493	0.4083
%Efficiency	100.00	95.07	59.17

Note : $[Fe^{2+}]$ = 5 mM, $[H_2O_2]$ = 15 mM, pH = 3 and Al_2O_3 = 74.07 g/l

Table D-16 The effects of initial hydrogen peroxide concentration on the 2,6-dimethylaniline oxidation in fluidized-bed reactor; COD removal

Time	COD removal, C/C_0		
min	2,6-DMA 0.5 mM	2,6-DMA 1 mM	2,6-DMA 5 mM
0	1.0000	1.0000	1.0000
2	0.9608	0.8583	0.8267
5	0.9412	0.7165	0.7867
10	0.9216	0.7165	0.7378
20	0.7843	0.7008	0.7111
40	0.7647	0.6535	0.6889
60	0.7451	0.6535	0.6356
90	0.7255	0.6220	0.6267
120	0.5882	0.5118	0.4667
150	0.5294	0.3858	0.3156
%Efficiency	47.0588	61.4173	68.4444

Note : $[Fe^{2+}]$ = 5 mM, $[H_2O_2]$ = 15 mM, pH = 3 and Al_2O_3 = 74.07 g/l

Table D-17 The effects of initial hydrogen peroxide concentration on the 2,6-dimethylaniline oxidation in fluidized-bed reactor; Total iron removal

Time	Total iron removal, C/C_0		
min	2,6-DMA 0.5 mM	2,6-DMA 1 mM	2,6-DMA 5 mM
0	1.0000	1.0000	1.0000
2	0.9050	0.9120	0.9380
5	0.8540	0.8970	0.8990
10	0.8170	0.8530	0.8620
20	0.7450	0.8110	0.8220
40	0.6830	0.7570	0.7640
60	0.6830	0.7390	0.7560
90	0.6410	0.7120	0.7470
120	0.6140	0.7220	0.7130
150	0.6150	0.7200	0.7350
%Efficiency	38.5350	27.9610	26.4630

Note : $[Fe^{2+}]$ = 5 mM, $[H_2O_2]$ = 15 mM, pH = 3 and Al_2O_3 = 74.07 g/l

Table D-18 The effects of initial hydrogen peroxide concentration on the 2,6-dimethylaniline oxidation in fluidized-bed reactor; Ferrous ion removal

Time	2,6-DMA removal, C/C_0		
min	2,6-DMA 0.5 mM	2,6-DMA 1 mM	2,6-DMA 5 mM
0	1.0000	1.0000	1.0000
2	0.9130	0.8590	0.7180
5	0.7050	0.6860	0.5260
10	0.5520	0.5020	0.4190
20	0.3920	0.3770	0.2930
40	0.2400	0.2190	0.1750
60	0.1910	0.1650	0.1710
90	0.1510	0.1470	0.1450
120	0.1370	0.1330	0.1560
150	0.1330	0.1170	0.1530
%Efficiency	86.6660	88.3170	84.6810

Note : $[Fe^{2+}]$ = 5 mM, $[H_2O_2]$ = 15 mM, pH = 3 and Al_2O_3 = 74.07 g/l

Table D-19 The effects of initial hydrogen peroxide concentration on the 2,6-dimethylaniline oxidation in fluidized-bed reactor; Hydrogen peroxide depletion

Time	H_2O_2 depletion, C/C_0		
min	2,6-DMA 0.5 mM	2,6-DMA 1 mM	2,6-DMA 5 mM
0	1.0000	1.0000	1.0000
2	0.0000	0.0000	0.0000
5	0.0000	0.0000	0.0000
10	0.0000	0.0000	0.0000
20	0.0000	0.0000	0.0000
40	0.0000	0.0000	0.0000
60	0.0000	0.0000	0.0000
90	0.0000	0.0000	0.0000
120	0.0000	0.0000	0.0000
150	0.0000	0.0000	0.0000
%Efficiency	100	100	100

Note : $[Fe^{2+}]$ = 5 mM, $[H_2O_2]$ = 15 mM, pH = 3 and Al_2O_3 = 74.07 g/l

(D-7) Comparison between fluidized-bed Fenton and conventional Fenton processes

Table D-20 Comparison between fluidized-bed Fenton and conventional Fenton processes onto the 2,6-dimethylanline oxidation

Time	2,6-DMA removal, C/C_0	
min	FB-Fenton	Fenton
0	1.0000	1.0000
2	0.1703	0.2519
5	0.1133	0.2133
10	0.1017	0.2029
20	0.0740	0.1750
40	0.0505	0.1709
60	0.0279	0.1671
90	0.0197	0.1292
120	0.0069	0.1127
150	0.0000	0.1146
%Efficiency	100.0000	88.5402

Note : [2,6-DMA] = 1 mM, $[Fe^{2+}]$ = 2.5 mM, $[H_2O_2]$ = 10 mM, pH = 3 and Al_2O_3 = 74.07 g/l

Table D-21 Comparison between fluidized-bed Fenton and conventional Fenton processes onto the total iron removal

Time	Total iron removal, C/C_0	
min	FB-Fenton	Fenton
0	1.0000	1.0000
2	0.9510	0.9876
5	0.9140	0.9848
10	0.8930	0.9757
20	0.8910	0.9736
40	0.8550	0.9580
60	0.8510	0.9419
90	0.8300	0.9341
120	0.7580	0.9279
150	0.7530	0.9213
%Efficiency	24.6740	7.8666

Note : [2,6-DMA] = 1 mM, $[Fe^{2+}]$ = 2.5 mM, $[H_2O_2]$ = 10 mM, pH = 3 and Al_2O_3 = 74.07 g/l

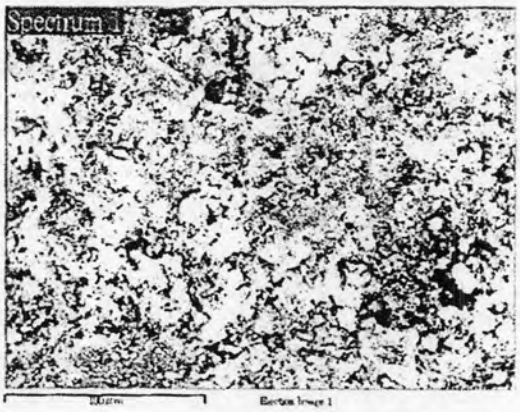
Table D-22 Comparison between fluidized-bed Fenton and conventional Fenton processes onto the ferrous ion removal

Time	Ferrous ion removal, C/C_0	
min	FB-Fenton	Fenton
0	1.000	1.0000
2	0.2072	0.8440
5	0.1281	0.6800
10	0.0619	0.5050
20	0.0206	0.3220
40	0.0151	0.2060
60	0.0195	0.1460
90	0.0328	0.1360
120	0.0284	0.1280
150	0.0518	0.1210
%Efficiency	94.8233	87.8728

Note : [2,6-DMA] = 1 mM, $[Fe^{2+}]$ = 2.5 mM, $[H_2O_2]$ = 10 mM, pH = 3 and Al_2O_3 = 74.07 g/l

APPENDIX E

Element	Weight%	Atomic%
O K	50.03	62.81
Al K	49.97	37.19
Totals	100.00	



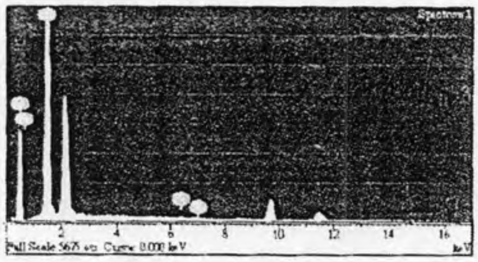
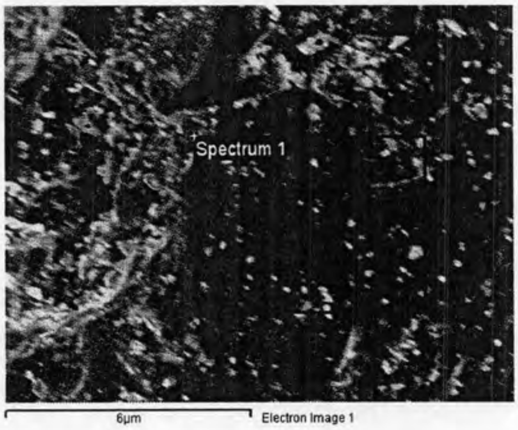


Figure E-1 XRD information, to investigate the element within new Al_2O_3

Element	Weight%	Atomic%
O K	36.61	52.08
Si K	57.61	46.69
Pd L	5.78	1.24
Totals	100.00	



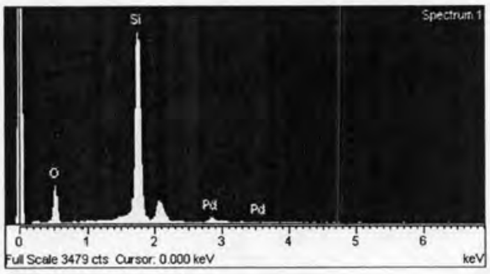
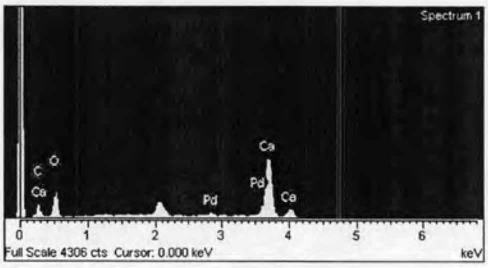


Figure E-2 XRD information, to investigate the element within new SiO_2

Element	Weight%	Atomic%
C K	9.43	16.46
O K	47.61	62.42
Ca K	38.75	20.28
Pd L	4.22	0.83
Totals	100.00	



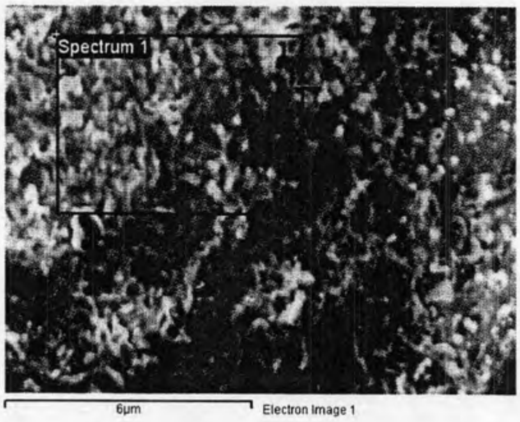
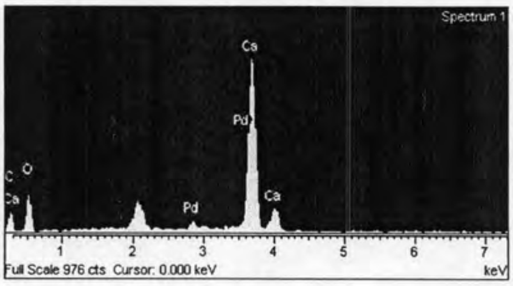


Figure E-3 XRD information, to investigate the element within new Black Gravel

Element	Weight%	Atomic%
C K	7.51	14.55
O K	38.08	55.42
Ca K	50.05	29.08
Pd L	4.36	0.95
Totals	100.00	



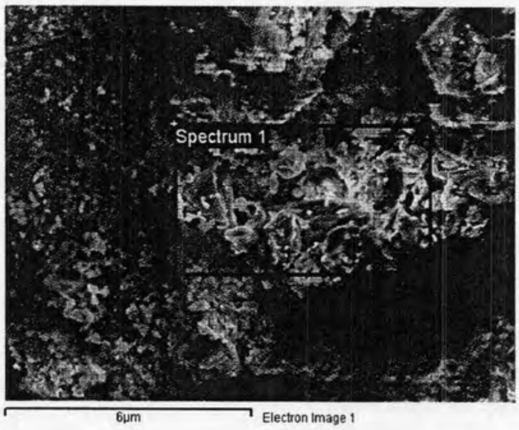
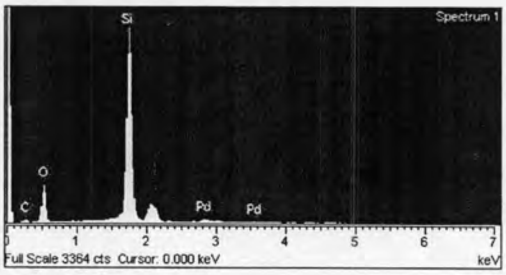


Figure E-4 XRD information, to investigate the element within new White Gravel

Element	Weight%	Atomic%
C K	12.59	20.84
O K	35.68	44.32
Si K	48.34	34.20
Pd L	3.39	0.63
Totals	100.00	



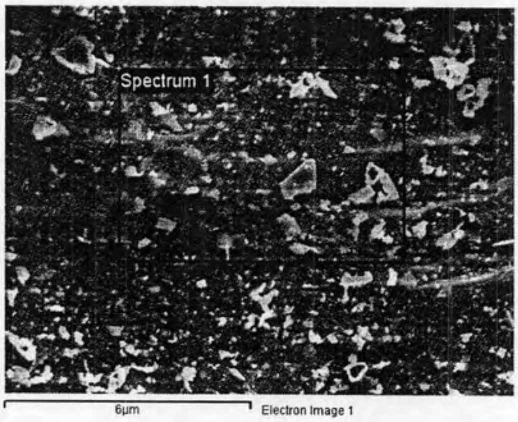
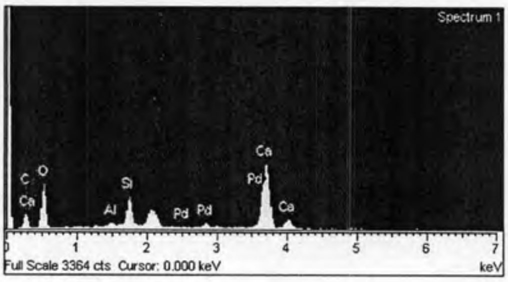


Figure E-5 XRD information, to investigate the element within new Brown Gravel

Element	Weight%	Atomic%
C K	11.66	19.21
O K	49.23	60.88
Al K	0.62	0.46
Si K	6.37	4.49
Ca K	29.22	14.43
Pd L	2.90	0.54
Totals	100.00	



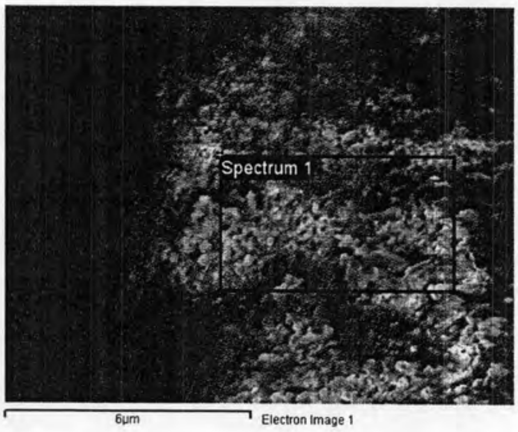


Figure E-6 XRD information, to investigate the element within new Colour Gravel

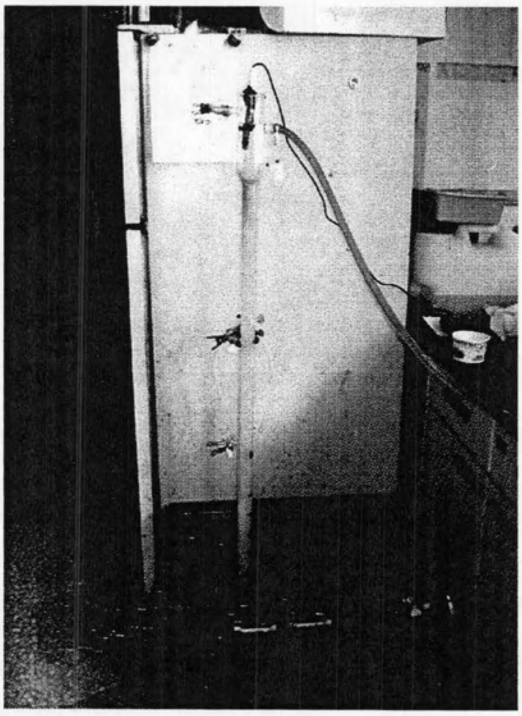


Figure E-8 Fluidized-bed reactor

BIOGRAPHY

Miss Sutthinee Narkwittaya was born on May 26, 1984 in Bangkok, Thailand. She graduated Bachelor's degree in Environmental Resource Chemistry from the faculty of Science, King Mongkut's Institute of Technology Ladkrabang 2006. After that, she pursued her Master's degree in Environmental Engineering from the faculty of Engineering, Chulalongkorn University on May, 2006. She had attended to oral presentation and submitted a publication with her principal advisor, Associate Professor Chavalit Ratanatamskul, Ph.D. and co-advisor, Professor Ming-Chun Lu, Ph.D., in the subject of "Oxidation of 2,6-dimethylaniline by fluidized-bed Fenton process", 2008 International Conference on Environmental Quality Concern, Control and Conservation on May 23, 2008, Tainan, Taiwan.