Supporting information

Visible-light-induced Photo-Fenton process for the facile degradation of metronidazole by Fe/Si codoped TiO_2

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Table S1 Application of doped/modified TiO_2 for photo-fenton reactions

Catalysts /amount	Pollutants/ initial amount	pH	light source	Degration time	Degradation efficiency %	ref
Fe-N-Ag-TiO_2/ 250 mg/L	Cephalexin 50 mg/L	3.5	Sunlight	150	90	[1]
mp-MXene/ TiO_{2-x}[a] NDs 13.3 mg/L	Rhodamine B 30 mg/L	5.78	500 W Xe lamp	10	96	[2]
Fe-TiO_2 1 mg/L	Reactive Brilliant Red X3B	3.0	300 W Xe lamp	20	96	[3]
Diatomite (Kieselguhr)-Fe_2O_3-TiO_2 2000 mg/L	0.16 mM methylene blue $2.9*10^{-5}$ mg/L	7.0	300 W Xe lamp	120	96	[4]
TiO_2/Fe_2TiO_5/ Fe_2O_3 1000 mg/L	Phenol 10 mg/L	4.0	300 W Xe lamp	60	Almost 100	[5]
SiO_2/Fe_3O_4/ C@TiO_2 250 mg/L	p-nitrophenol 8 g/L	3.0	500W UV lamp	240	90	[6]
Fe_3O_4@void @TiO_2 250 mg/L	Tetracycline 40 mg/L	7.0	300 W Xe lamp	10	95	[7]
Fe/Si codoped TiO_2 1000 mg/L	MNZ 10 mg/L	7.0	220W Xe lamp	50	93	This work

[a] microporous MXene monolayers embedded with Ti^{3+}-doped TiO2 nanodots

The experimental design (or design of experiments) in optimization of MNZ degradation efficiency is also a vital part of the experiment. It has been reported that while analyzing the effects of multiple process parameters on one or more responses the use of conventional methods like one variable at a time (OVAT) could not be applied, as these were time consuming and labor intensive. Therefore, the use of suitable statistical approach which could reduce the number of experiments and offer reasonably reliable results were proposed in such situations. In this study, a statistical multivariate approach using BBD (Box-Behnken Design) in combination with RSM (Response Surface Methodology) was adopted to find the optimization condition [8]. All the statistical analyses and plots were made using the Design-Expert software (version 8.0.7.1)

The relationship between the response (Y) and process parameters (X_i) as represented by the response function as given in **equation (1)**

$$Y= b_0 + b_1X_1 + b_2X_2 + b_3X_3 + b_4X_4 + b_5X_5 + b_6X_6 + b_{11}X_1^2 + b_{12}X_1X_2 + b_{13}X_1X_3 + b_{14}X_1X_4 + b_{15}X_1X_5 + b_{16}X_1X_6 + b_{22}X_{22} + b_{23}X_2X_3 + b_{24}X_2X_4 + b_{25}X_2X_5 + b_{26}X_2X_6 + b_{33}X_3^2 + b_{34}X_3X_4 + b_{35}X_3X_5 + b_{36}X_3X_6 + b_{44}X_4^2 + b_{45}X_4X_5 + B_{46}X_4X_6 + b_{55}X_5^2 + b_{56}X_5X_6 + b_{66}X_6^2 \quad \text{Eq. (1)}$$

Where: b_i depicts the response function coefficients for process parameters. The coded values for low, middle and high levels of the process parameters (concentration of MNZ, ppm, dosage of H_2O_2, mL, initial pH, dosage of Fe/Si codoped TiO_2, g, degradation time, min, the ratio of Fe and Si) are shown in **Table S2**.

Table S2 Coded representation of process parameters for statistical analysis.

			Coded Valves			
No.	Process Parameters	Units	Symbolic representation	-1 (Low)	0 (Middle)	+1 (High)
1	MNZ	ppm	X_1	1	5.5	10
2	H_2O_2	mL	X_2	0.1	0.4	0.7
3	pH	~	X_3	2	4.5	7
4	amount of Fe/Si codopedTiO_2	g	X_4	0.05	0.17	0.3
5	time	min	X_5	20	45	70
6	Fe:Si	~	X_6	1	3.5	6

Adequacy of the designed models was assessed using "goodness of fit" and analysis of variance (ANOVA) technique. RSM was used to generate 3D surface plots from the validated models. These surface plots were used to locate the optimum points of process parameters to attain maximum degradation of MNZ.

The response functions generated for Y, % MNZ degradation using the equation (1) was presented in **equation (2)**

$$Y= -53.15 - 6.87X_1 - 31.71X_2 + 35.03X_3 - 78.44X_4 + 0.74X_5 + 1.36X_6 + 4.39X_1X_2 - 0.58X_1X_3 + 13.20X_1X_4 - 0.03X_1X_5 + 0.20X_1X_6 - 1.39X_2X_3 + 34.83X_3X_4 + 0.05X_2X_5 - 1.78X_2X_6 + 22.39X_3X_4 - 0.14X_3X_5 + 1.50X_3X_6 + 0.36X_4X_5 + 10.04X4X6 + 0.08X_5X_6 + 0.38X_1^2 + 16.27X_2^2 - 2.90X_3^2 - 116.58X_4^2 - 1.11X_5^2 - 1.18X_6^2$$

Eq. (2)

All the terms are included in the equations irrespective of their statistical significance. The experimental responses (results of 54 BBD experimental set) is shown in **Table S3**

Table S3 Experimental responses with different variables

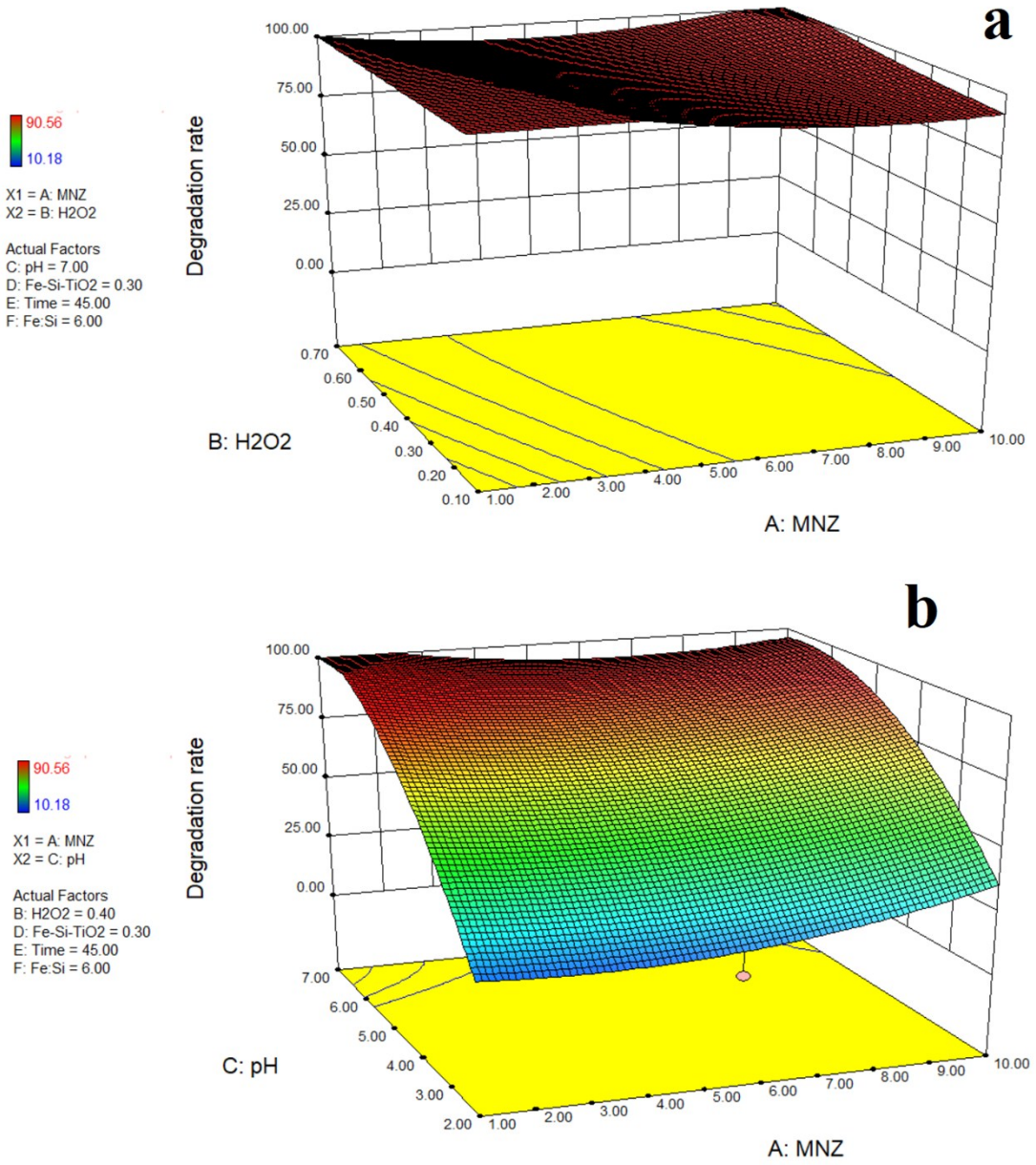
	variables						experimental responses
No.	MNZ coded valves (ppm)	H_2O_2 coded valves (mL)	pH coded valves	Fe/Si codoped TiO2 coded valves (g)	time coded valves (min)	Fe:Si coded valves	Y (%)
1	0 5.5	+1 0.7	+1 7.0	0 0.17	+1 70	0 3.5	43.38
2	0 5.5	0 0.4	-1 2.0	+1 0.30	0 45	-1 1	10.21
3	0 5.5	-1 0.1	+1 7.0	0 0.17	+1 70	0 3.5	41.23
4	1 10	0 0.4	-1 2.0	0 0.17	0 45	-1 1	10.23
5	0 5.5	0 0.4	0 4.5	0 0.17	0 45	0 3.5	65.51
6	0 5.5	0 0.4	0 4.5	0 0.17	0 45	0 3.5	68.67
7	0 5.5	+1 0.7	0 4.5	0 0.17	+1 70	-1 1	43.97
8	-1 1	0 0.4	0 4.5	+1 0.30	-1 20	0 3.5	77.67
9	0 5.5	-1 0.1	-1 2.0	0 0.17	-1 20	0 3.5	10.32
10	-1 1	0 0.4	0 4.5	-1 0.05	-1 20	0 3.5	30.1
11	0 5.5	+1 0.7	-1 2.0	0 0.17	-1 20	0 3.5	10.25
12	-1 1	-1 0.1	0 4.5	-1 0.05	0 45	0 3.5	72.41
13	-1 1	0 0.4	0 4.5	+1 0.30	+1 70	0 3.5	85.44
14	1 10	+1 0.7	0 4.5	-1 0.05	0 45	0 3.5	27.32
15	-1 1	0 0.4	+1 7.0	0 0.17	0 45	+1 6	84.93
16	0 5.5	-1 0.1	+1 7.0	0 0.17	-1 20	0 3.5	81.85
17	0 5.5	0 0.4	-1 2.0	-1 0.05	0 45	-1 1	10.18

18	1 10	-1 0.1	0 4.5	-1 0.05	0 45	0 3.5	14.02
19	1 10	0 0.4	0 4.5	0 0.17	0 45	-1 1	23.2
20	0 5.5	0 0.4	+1 7.0	+1 0.30	0 45	+1 6	79.43
21	1 10	0 0.4	0 4.5	0 0.17	0 45	+1 6	67.81
22	0 5.5	-1 0.1	0 4.5	0 0.17	-1 20	+1 6	38.49
23	-1 1	0 0.4	0 4.5	-1 0.05	+1 70	0 3.5	66.02
24	0 5.5	0 0.4	+1 7.0	+1 0.30	0 45	-1 1	27.53
25	0 5.5	-1 0.1	-1 2.0	0 0.17	+1 70	0 3.5	10.25
26	-1 1	+1 0.7	0 4.5	-1 0.05	0 45	0 3.5	56.31
27	1 10	+1 0.7	0 4.5	+1 0.30	0 45	0 3.5	82.43
28	0 5.5	0 0.4	-1 2.0	+1 0.30	0 45	+1 6	10.21
29	-1 1	-1 0.1	0 4.5	+1 0.30	0 45	0 3.5	79.31
30	0 5.5	0 0.4	0 4.5	0 0.17	0 45	0 3.5	55.02
31	1 10	0 0.4	-1 2.0	0 0.17	0 45	+1 6	20
32	0 5.5	0 0.4	0 4.5	0 0.17	0 45	0 3.5	10.22
33	0 5.5	0 0.4	+1 7.0	-1 0.05	0 45	-1 1	12.09
34	1 10	0 0.4	0 4.5	-1 0.05	+1 70	0 3.5	20.73
35	-1 1	+1 0.7	0 4.5	+1 0.30	0 45	0 3.5	74.13
36	-1 1	0 0.4	-1 2.0	0 0.17	0 45	-1 1	10.23
37	1 10	0 0.4	0 4.5	-1 0.05	-1 20	0 3.5	28.86
38	0 5.5	0 0.4	0 4.5	0 0.17	0 45	0 3.5	54.86
39	0 5.5	-1 0.1	0 4.5	0 0.17	+1 70	-1 1	35.31

40	0 5.5	0 0.4	0 4.5	0 0.17	0 45	0 3.5	58.62
41	1 10	0 0.4	0 4.5	+1 0.30	-1 20	0 3.5	60.2
42	0 5.5	-1 0.1	0 4.5	0 0.17	-1 20	-1 1	10.41
43	0 5.5	+1 0.7	-1 2.0	0 0.17	+1 70	0 3.5	10.19
44	0 5.5	-1 0.1	0 4.5	0 0.17	+1 70	+1 6	94.56
45	1 10	0 0.4	0 4.5	+1 0.30	+1 70	0 3.5	89.22
46	0 5.5	+1 0.7	+1 7.0	0 0.17	-1 20	0 3.5	71.38
47	0 5.5	+1 0.7	0 4.5	0 0.17	+1 70	+1 6	85.99
48	0 5.5	+1 0.7	0 4.5	0 0.17	-1 20	-1 1	14.25
49	0 5.5	0 0.4	0 4.5	-1 0.05	0 45	+1 6	38.89
50	1 10	-1 0.1	0 4.5	+1 0.30	0 45	0 3.5	69.6
51	0 5.5	+1 0.7	0 4.5	0 0.17	-1 20	+1 6	44.89
52	0 5.5	0 0.4	-1 2.0	-1 0.05	0 45	+1 6	10.25
53	-1 1	0 0.4	+1 7.0	0 0.17	0 45	-1 1	48.05
54	-1 1	0 0.4	-1 2.0	0 0.17	0 45	+1 6	10.23

Using Design-Expert software, 3-dimensional surface plots with contour plots were generated to find the optimal conditions of the process parameters. These plots are shown in **Fig. S1 (a)-(o)**:

From the surface plots, the maximum degradation levels of MNZ at optimum conditions of process parameters were obtained. The maximum degradation of MNZ is observed to be 94.56%.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10.18
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X2 = B: H2O2
Actual Factors
C: pH = 7.00
D: Fe-Si-TiO2 = 0.30
E: Time = 45.00
F: Fe:Si = 6.00
Degradation rate
100.00
75.00
50.00
25.00
0.00
B: H2O2
0.70
0.60
0.50
0.40
0.30
0.20
0.10
A: MNZ
1.00
2.00
3.00
4.00
5.00
6.00
7.00
8.00
9.00
10.00
b
90.56
10.18
X1 = A: MNZ
X2 = C: pH
Actual Factors
B: H2O2 = 0.40
D: Fe-Si-TiO2 = 0.30
E: Time = 45.00
F: Fe:Si = 6.00
Degradation rate
100.00
75.00
50.00
25.00
0.00
C: pH
7.00
6.00
5.00
4.00
3.00
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A: MNZ
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7.00
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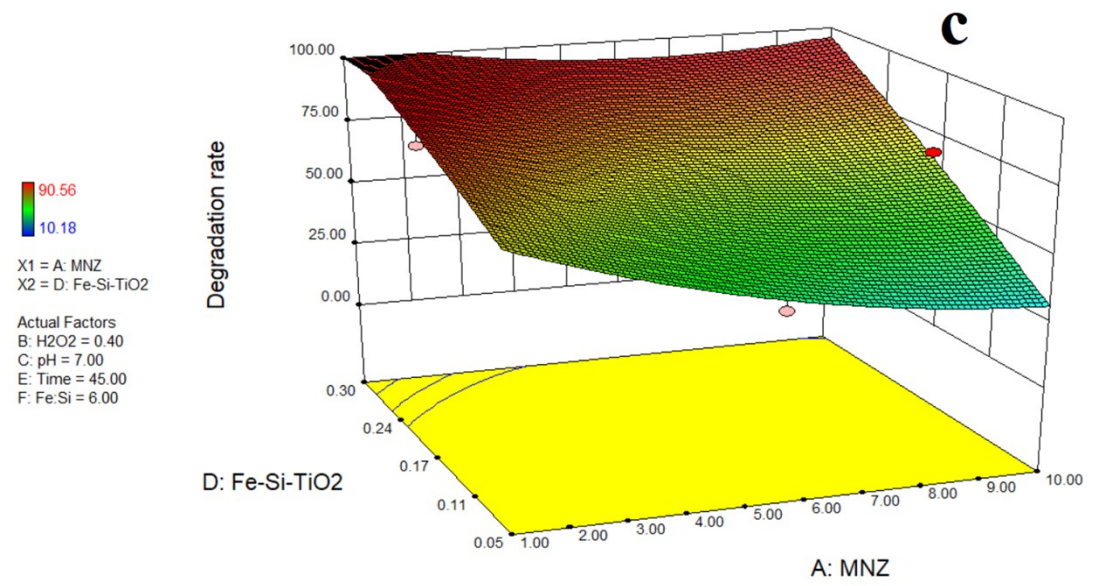
10.00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X2 = D: Fe-Si-TiO2
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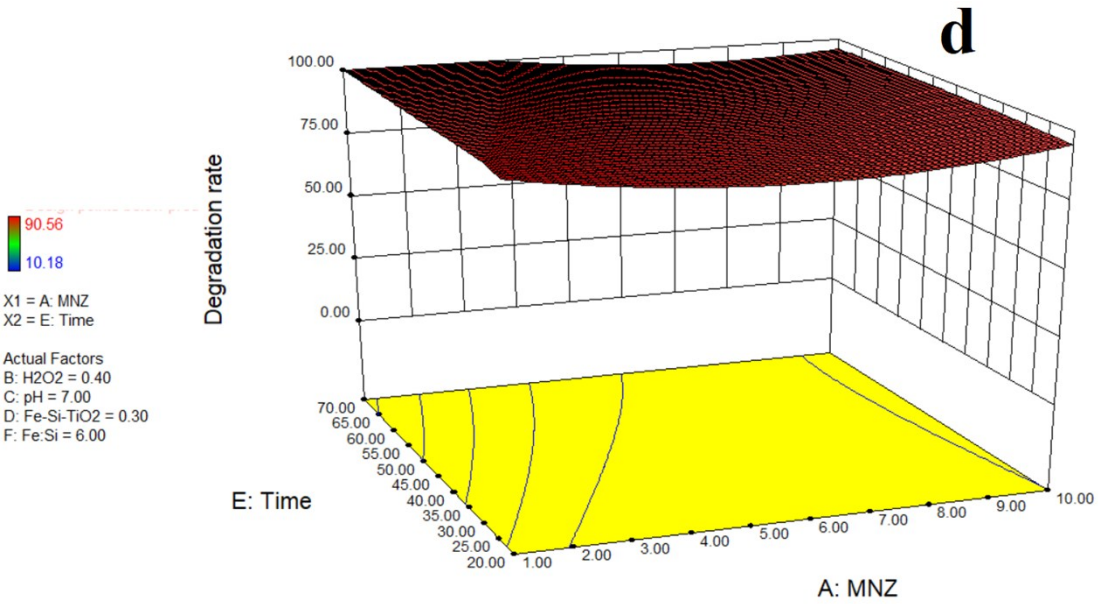
C: pH = 7.00
E: Time = 45.00
F: Fe:Si = 6.00
Degradation rate
100.00
75.00
50.00
25.00
0.00
D: Fe-Si-TiO2
0.30
0.24
0.17
0.11
0.05
A: MNZ
1.00
2.00
3.00
4.00
5.00
6.00
7.00
8.00
9.00
10.00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X2 = E: Time
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C: pH = 7.00
D: Fe-Si-TiO2 = 0.30
F: Fe:Si = 6.00
Degradation rate
100.00
75.00
50.00
25.00
0.00
E: Time
70.00
65.00
60.00
55.00
50.00
45.00
40.00
35.00
30.00
25.00
20.00
A: MNZ
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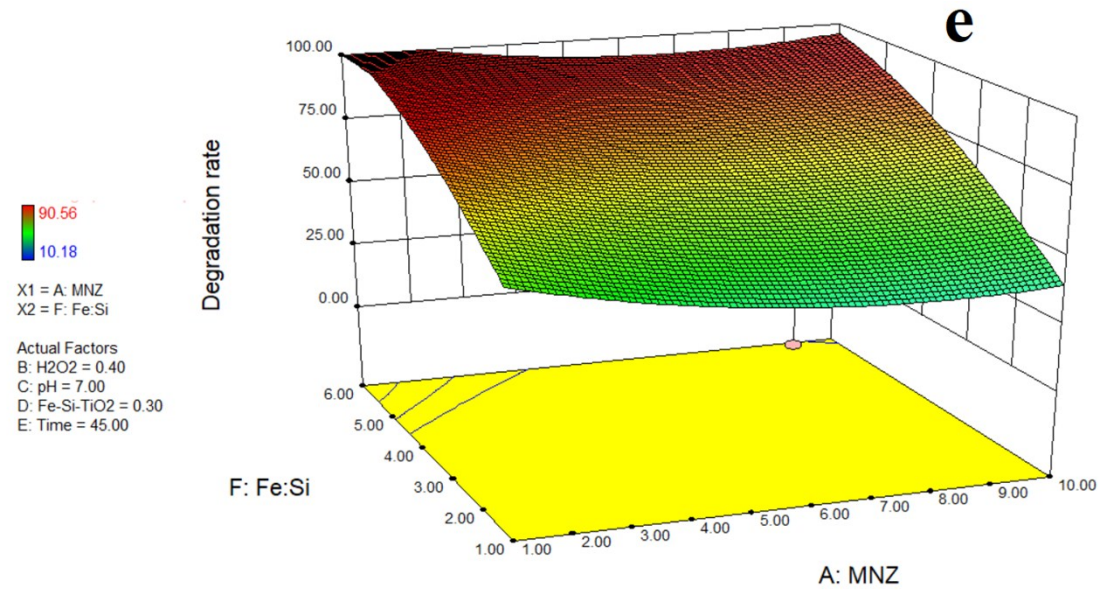
5.00
6.00
7.00
8.00
9.00
10.00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10.18
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X2 = F: Fe:Si
Actual Factors
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C: pH = 7.00
D: Fe-Si-TiO2 = 0.30
E: Time = 45.00
Degradation rate
100.00
75.00
50.00
25.00
0.00
F: Fe:Si
6.00
5.00
4.00
3.00
2.00
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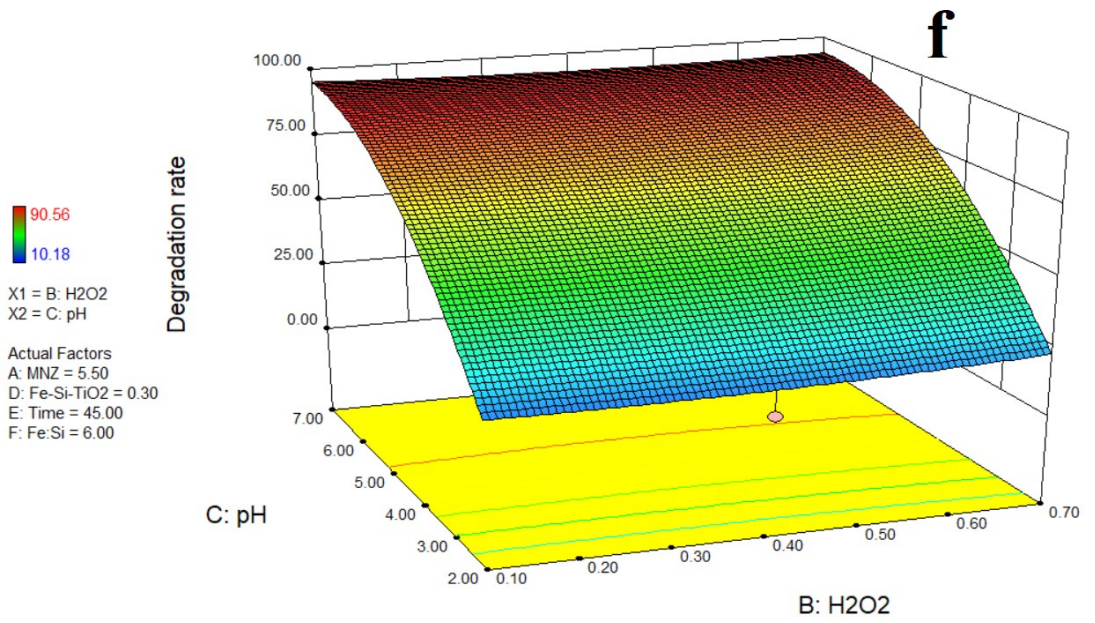
5.00
6.00
7.00
8.00
9.00
10.00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10.18
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X2 = C: pH
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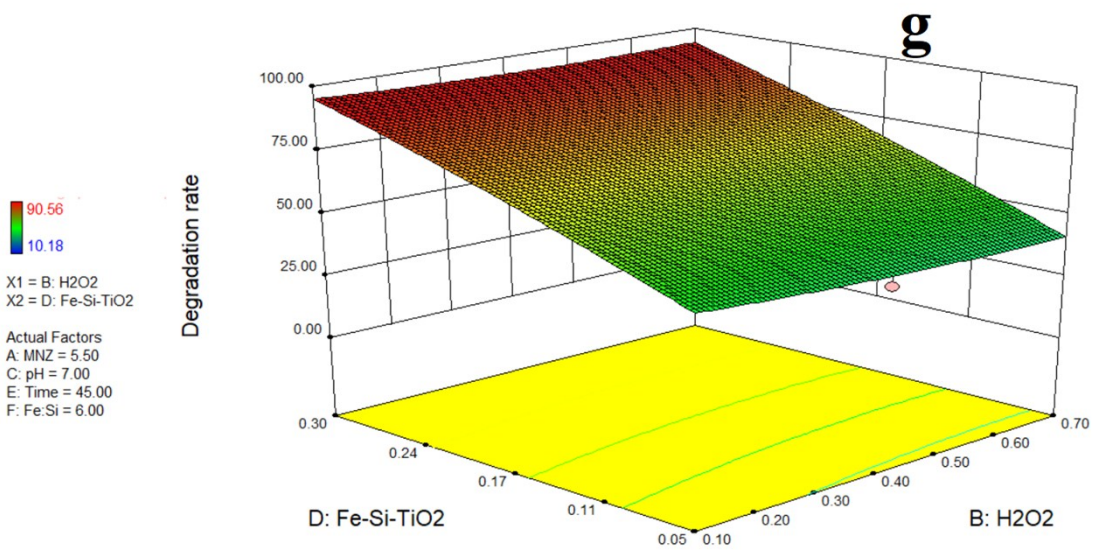
D: Fe-Si-TiO2 = 0.30
E: Time = 45.00
F: Fe:Si = 6.00
Degradation rate
100.00
75.00
50.00
25.00
0.00
7.00
6.00
5.00
4.00
3.00
2.00
C: pH
0.10
0.20
0.30
0.40
0.50
0.60
0.70
B: H2O2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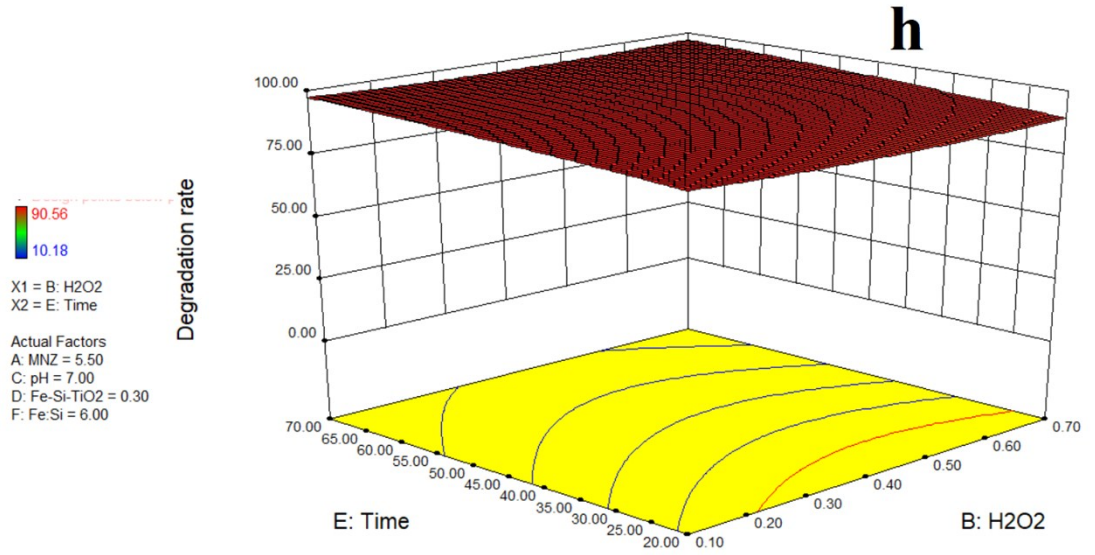
C: pH = 7.00
E: Time = 45.00
F: Fe:Si = 6.00
Degradation rate
100.00
75.00
50.00
25.00
0.00
0.30
0.24
0.17
0.11
0.05
D: Fe-Si-TiO2
0.10
0.20
0.30
0.40
0.50
0.60
0.70
B: H2O2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10.18
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X2 = E: Time
Actual Factors
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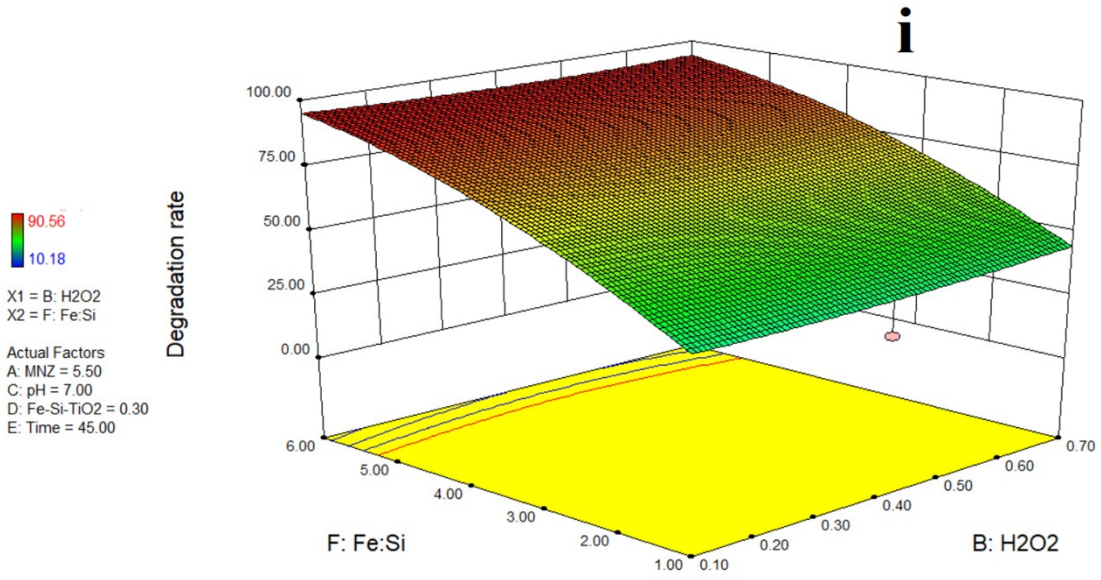
C: pH = 7.00
D: Fe-Si-TiO2 = 0.30
F: Fe:Si = 6.00
Degradation rate
100.00
75.00
50.00
25.00
0.00
70.00
65.00
60.00
55.00
50.00
45.00
40.00
35.00
30.00
25.00
20.00
E: Time
0.10
0.20
0.30
0.40
0.50
0.60
0.70
B: H2O2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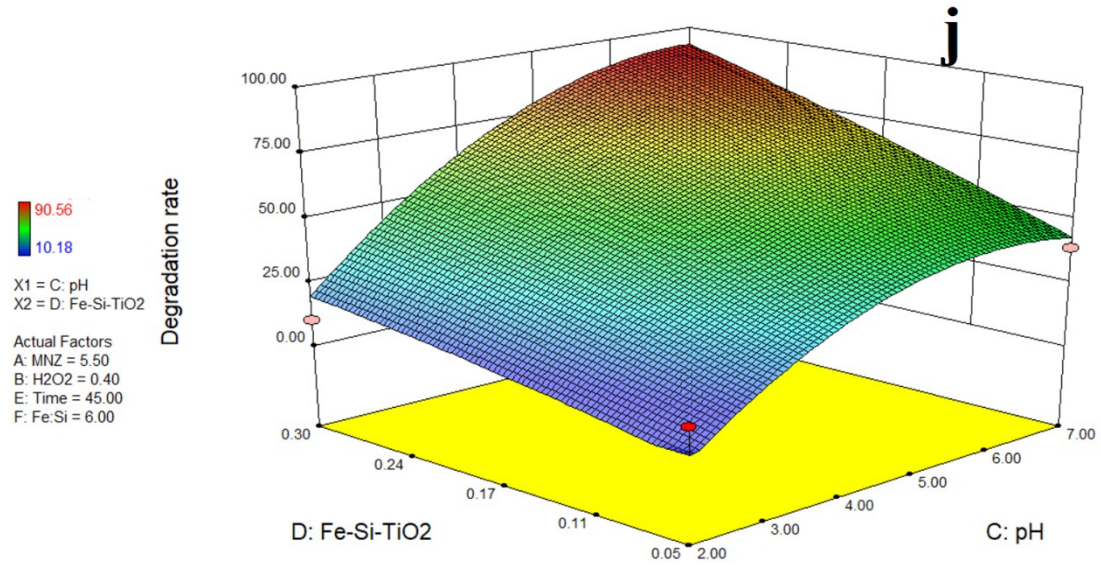
X2 = F: Fe:Si
Actual Factors
A: MNZ = 5.50
C: pH = 7.00
D: Fe-Si-TiO2 = 0.30
E: Time = 45.00
Degradation rate
100.00
75.00
50.00
25.00
0.00
6.00
5.00
4.00
3.00
2.00
1.00
F: Fe:Si
0.10
0.20
0.30
0.40
0.50
0.60
0.70
B: H2O2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X2 = D: Fe-Si-TiO2
Actual Factors
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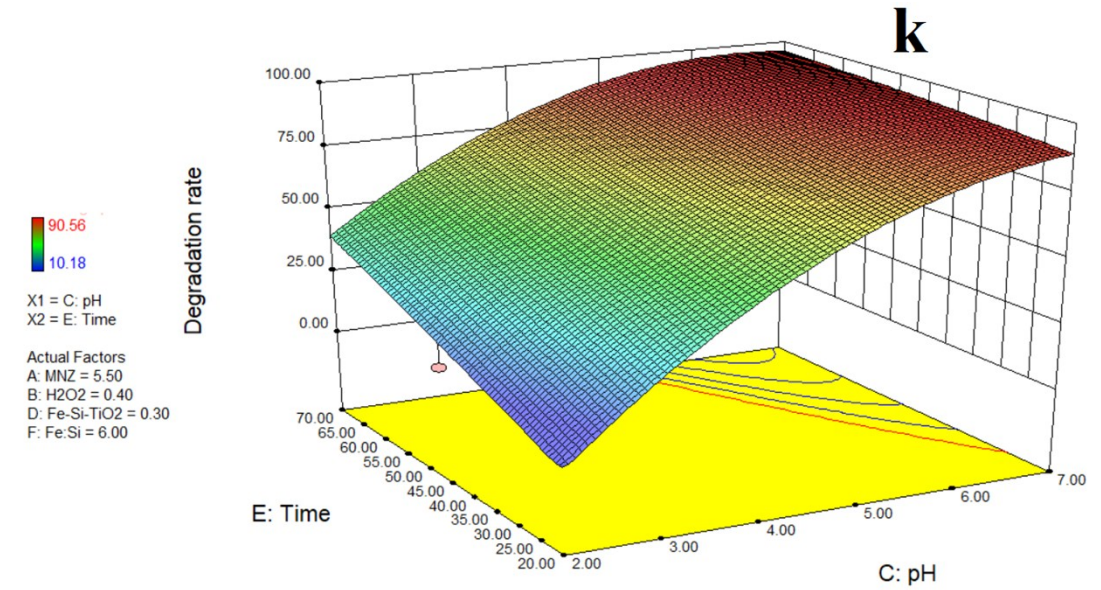
B: H2O2 = 0.40
E: Time = 45.00
F: Fe:Si = 6.00
Degradation rate
100.00
75.00
50.00
25.00
0.00
0.30
0.24
0.17
0.11
0.05
D: Fe-Si-TiO2
2.00
3.00
4.00
5.00
6.00
7.00
C: pH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10.18
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X2 = E: Time
Actual Factors
A: MNZ = 5.50
B: H2O2 = 0.40
D: Fe-Si-TiO2 = 0.30
F: Fe:Si = 6.00
Degradation rate
100.00
75.00
50.00
25.00
0.00
70.00
65.00
60.00
55.00
50.00
45.00
40.00
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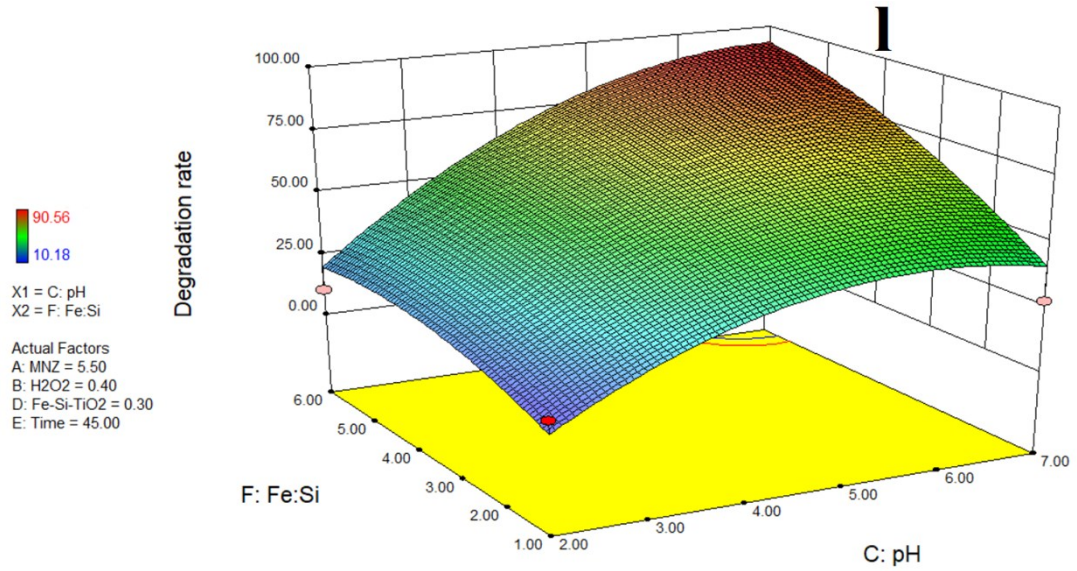
4.00
5.00
6.00
7.00
C: pH

l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X2 = F: Fe:Si
Actual Factors
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B: H2O2 = 0.40
D: Fe-Si-TiO2 = 0.30
E: Time = 45.00
Degradation rate
100.00
75.00
50.00
25.00
0.00
6.00
5.00
4.00
3.00
2.00
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F: Fe:Si
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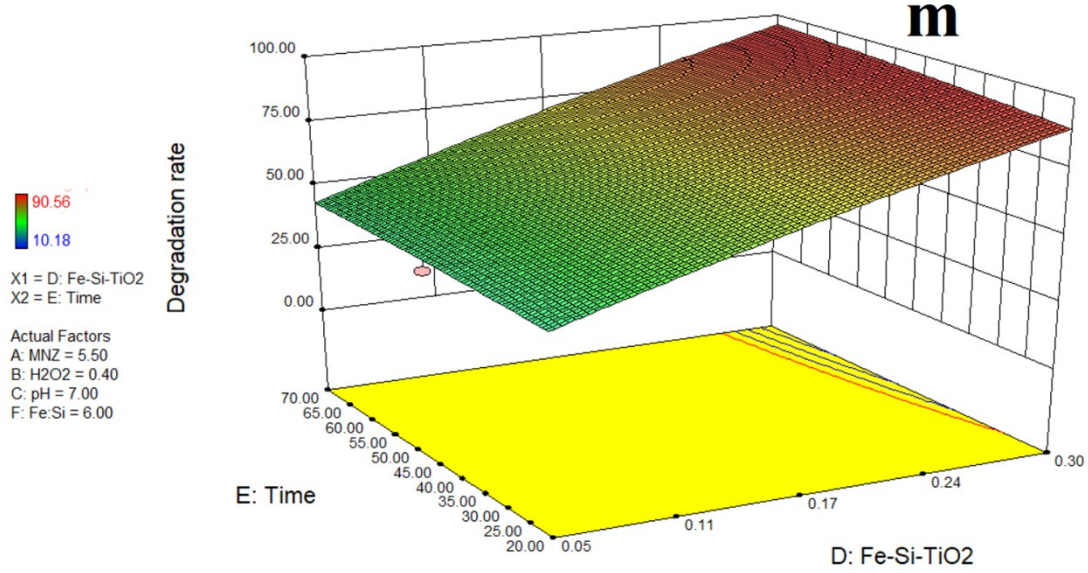
4.00
5.00
6.00
7.00
C: pH

m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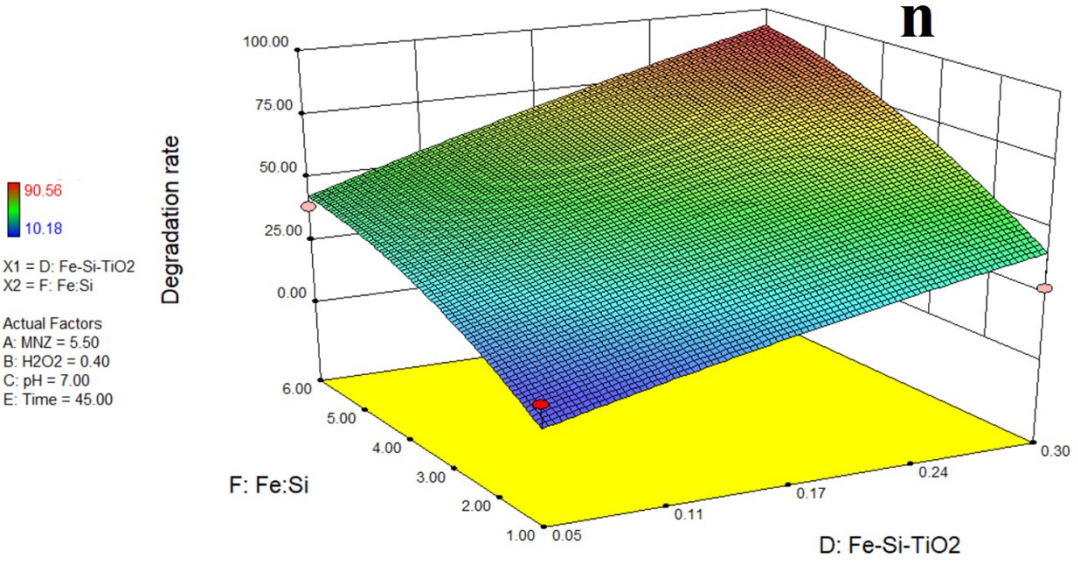
X2 = E: Time
Actual Factors
A: MNZ = 5.50
B: H2O2 = 0.40
C: pH = 7.00
F: Fe:Si = 6.00
Degradation rate
100.00
75.00
50.00
25.00
0.00
70.00
65.00
60.00
55.00
50.00
45.00
40.00
35.00
30.00
25.00
20.00
E: Time
0.05
0.11
0.17
0.24
0.30
D: Fe-Si-TiO2

n


90.56
10.18
X1 = D: Fe-Si-TiO2
X2 = F: Fe:Si
Actual Factors
A: MNZ = 5.50
B: H2O2 = 0.40
C: pH = 7.00
E: Time = 45.00
Degradation rate
100.00
75.00
50.00
25.00
0.00
6.00
5.00
4.00
3.00
2.00
1.00
F: Fe:Si
0.05
0.11
0.17
0.24
0.30
D: Fe-Si-TiO2

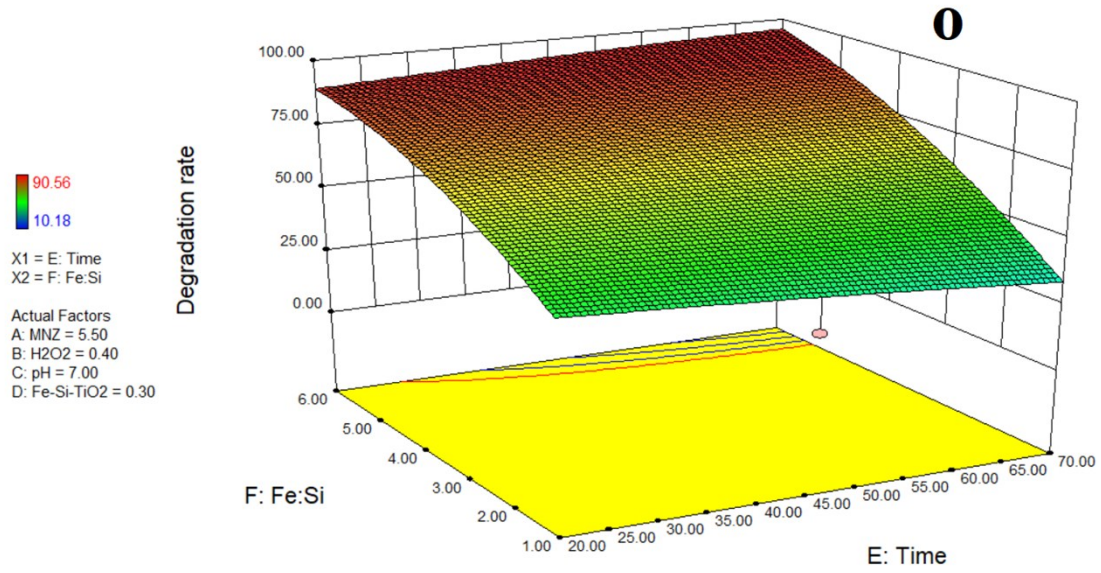


Fig. S1 Surface plots: concentration of MNZ and dosage of H_2O_2(a), concentration of MNZ and pH(b), concentration of MNZ and dosage of catalyst(c), concentration of MNZ and time(d), concentration of MNZ and molar ratio of Fe/Si(e), dosage of H_2O_2 and pH(f), dosage of H_2O_2 and dosage of catalyst(g), dosage of H_2O_2 and time(h), dosage of H_2O_2 and molar ratio of Fe/Si(i), dosage of catalyst and pH(j), time and pH(k), molar ratio of Fe/Si and pH(l), dosage of catalyst and time(m), dosage of catalyst and molar ratio of Fe/Si(n), time and molar ratio of Fe/Si(o), the ordinate was the MNZ degradation efficiency (%).

The process of Fe/Si codoped TiO_2 for degradation of MNZ contaminated water under visible light illumination in the presence of H_2O_2 was also optimized using multivariate BBD approach in combination with RSM (Fig. S1 f-i). The degradation rate of MNZ was not increased obviously by increasing of the dosage of H_2O_2. For this Photo-Fenton system, H_2O_2 was more likely to be an inducer, which assisted Fe^{3+} to catch e^- more quickly. According to the optimization by RSM, the most suitable process conditions for the best degradation rate were as follows (the purpose was less time, high degradation efficiency, low cost of consumption), the concentration of MNZ: 6 ppm; the dosage of H_2O_2, 0.4 mL/300 mL (10.0 M); pH, 7.0; the dosage of catalysts, 0.3g; the degradation time, 50 min; and the ratio of Fe, Si, 6.0. It was expected to achieve 95% MNZ degraded approximately.

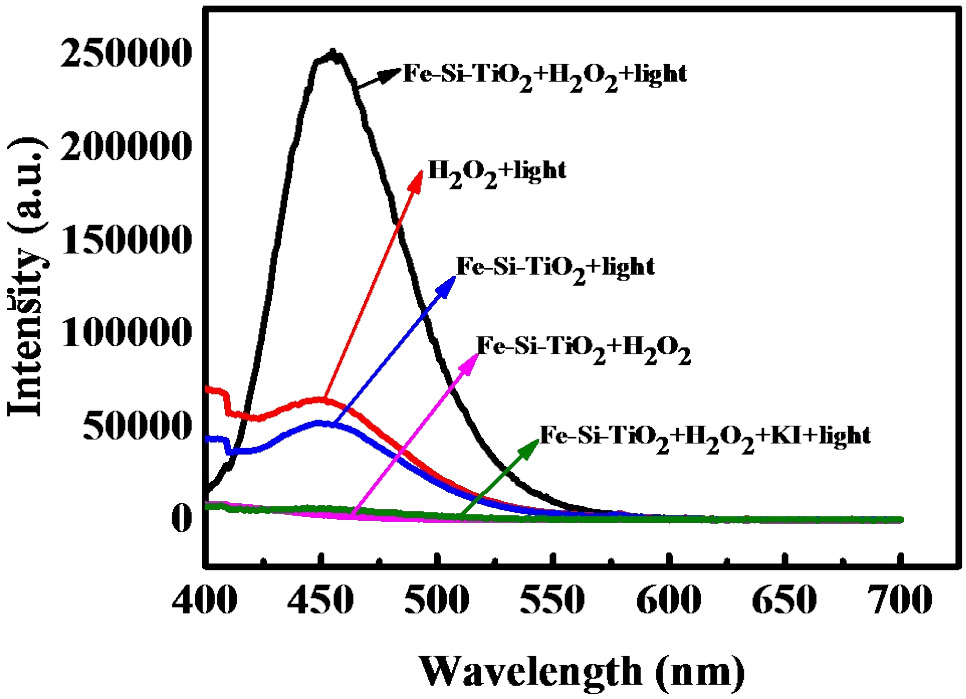


Fig. S2 Luminescence response of trimesic acid reacted with different catalysts under light illumination.

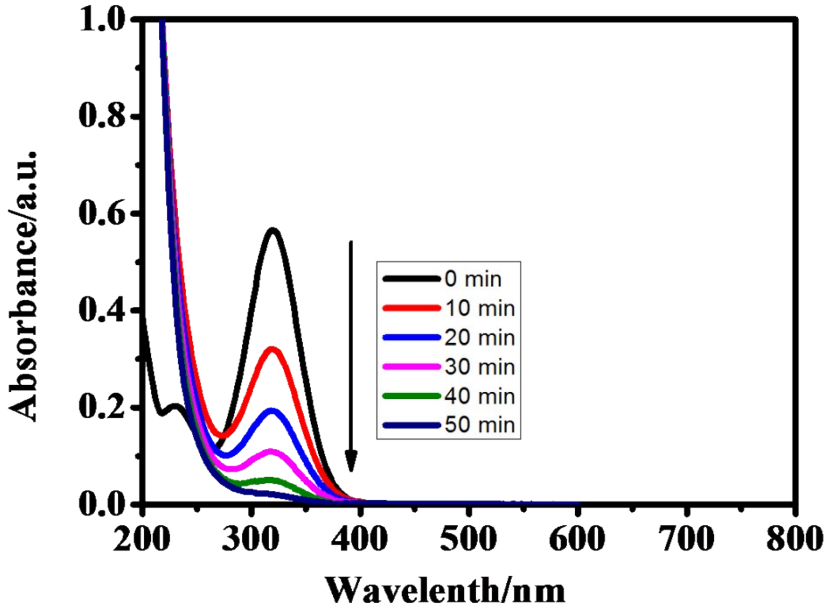


Fig. S3 UV-vis spectra of MNZ during the photo-fenton process.

To further confirm the stability of the Fe/Si codoped, the used catalyst after reaction was examined by HRTEM and XPS, the results are shown in **Fig. S2** and **Fig. S3**. Very tiny differences in the elemental compositions and chemical states of the iron species were observed for the fresh and used Fe/Si codoped TiO_2. All these results clearly reveal that the catalyst is very stable and can have a good catalytic activity.

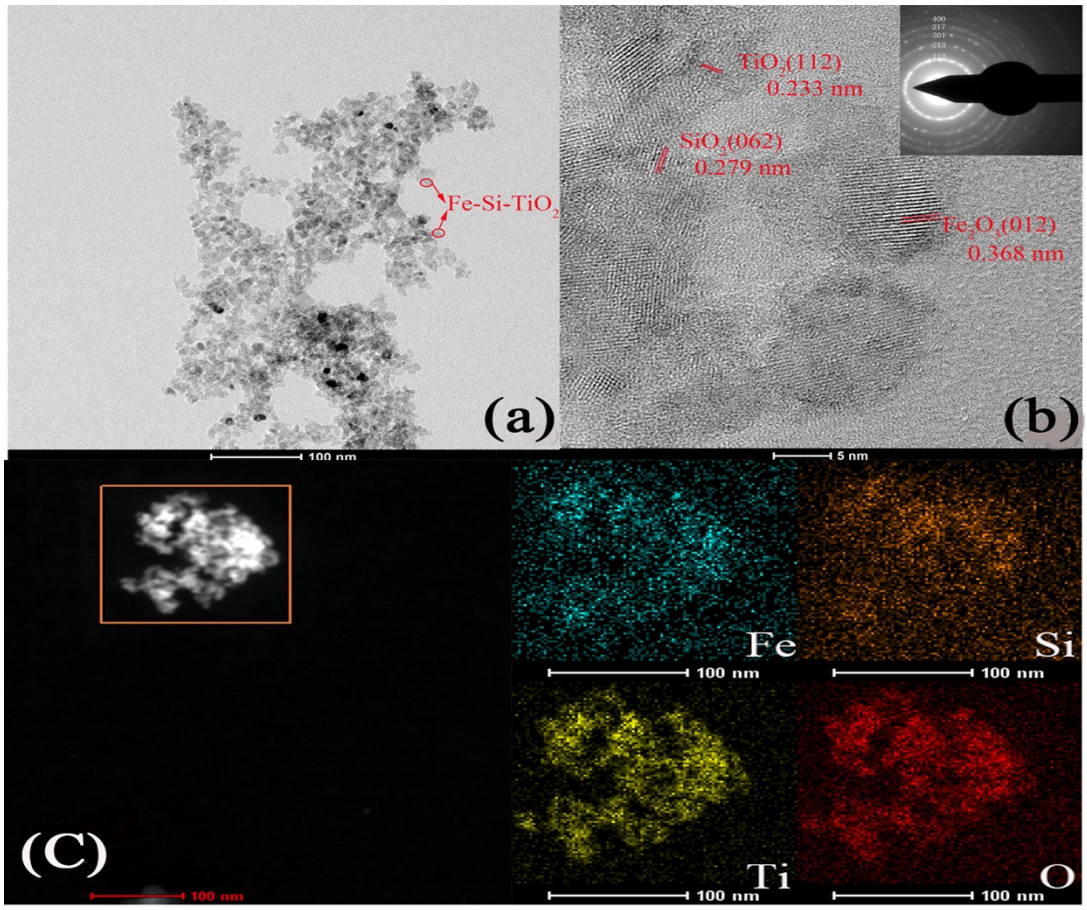


Fig. S4. FESTEM images of Fe/Si codoped TiO_2 after degradation, **Fig. S2a** was the image with low magnification figure and **Fig. S2b** was the image with high magnification**. Fig. S2 c:** Element mapping images of Fe/Si codoped TiO_2.

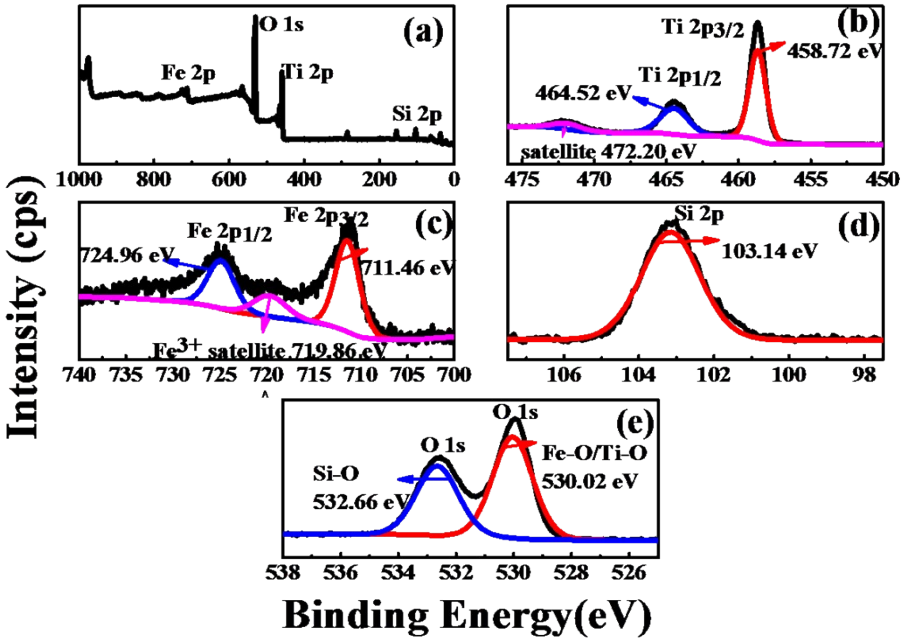


Fig. S5. XPS spectra of Fe/Si codoped TiO_2 after Photo-Fenton degradation reaction: (a) survey; (b) Ti 2p peaks; (c) Fe 2ppeaks; (d) Si 2p peak; (e) O 1s peaks.

Table S 4 MNZ degradation efficiency comparison by different catalyst under different conditions for 50 min.

catalystic system / methods	homogeneous reaction (Fe^{3+}+H_2O_2)	TiO_2	Si doped TiO_2	Fe doped TiO_2	Fe/Si codoped TiO_2 without calcination	Fe/Si codoped TiO_2 with calcinations treatment
fenton	\	1.39%	1.41%	1.44%	1.43%	1.51%
photo	\	5.46%	6.03%	20.65%	7.47%	22.26%
Photo-fenton	5.98%	5.54%	6.33%	79.39%	30.21%	93.04%

Table S 5 Comparison of MNZ degradation efficiencies by different Fenton, Photo-Fenton or photo systems.

Catalysts concentration	C_{MNZ} (mg/L)	Methods	Light source	pH	t (min)	Efficiency (%)	Active specie	K (min^{-1})	Ref
TiO_2 1 g/L	80	Photo	125W UV lamp	7.0	120	95	•OH	0.0233	9
$BiVO_4/FeVO_4$ 4 g/L	10	Photo	500 W Xe lamp	7.0	90	84	$•O_2^-$	\	10
FeS_2O_8 3 mM/L	500	Fenton	\	3.0	180	96	•OH	0.01	11
$BiVO_4/BiPO_4$ 0.5 g/L	5	Photo	300 W UV lamp	10.0	360	64.5	•OH/ $•O_2^-$	0.015	12
Nano ZnO 1.5 g/L	80	Photo	180 W UV lamp	7.0	180	96.5	•OH	\	13
Fe-SnO_2/Co_3O_4 2 g/L	30	Photo	125 W UV lamp	6.0	15	98.3	\	\	14
Fe/Si codoped TiO_2 1 g/L	10	Photo -Fenton	220 W Xe lamp	7.0	50	93	•OH	0.049	This work

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