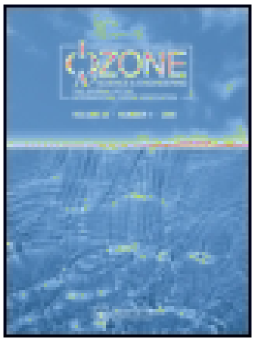

Ozone: Science & Engineering

The Journal of the International Ozone Association

ISSN: 0191-9512 (Print) 1547-6545 (Online) Journal homepage: http://www.tandfonline.com/loi/bose20

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To cite this article: Shengtao Jiang, Jianzhong Zhu, Ying Ding, Shuli Bai, Yujiang Guan & Jia Wang (2015): Degradation Effect and Mechanism of Dinitrotoluene Wastewater by Magnetic Nano-Fe_3O_4/H_2O_2 Fenton-like, Ozone: Science & Engineering, DOI: 10.1080/01919512.2015.1115716

OZONE: SCIENCE & ENGINEERING
http://dx.doi.org/10.1080/01919512.2015.1115716

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Degradation Effect and Mechanism of Dinitrotoluene Wastewater by Magnetic Nano-Fe_3O_4/H_2O_2 Fenton-like

Shengtao Jiang[a,b], Jianzhong Zhu[b], Ying Ding[b], Shuli Bai[a], Yujiang Guan[a], and Jia Wang[a]

[a]Zhejiang Provincial Key Laboratory of Plant Evolutionary Ecology and Conservation, Taizhou University, Tai Zhou City 318000, Zhejiang Province, P.R. China; [b]Key Laboratory for Integrated Regulation and Resource Development on Shallow Lake of Ministry of Education, Hohai University, Nanjing City 210098, Jiangsu Province, P.R. China

ABSTRACT

The characteristics and influencing factors for dinitrotoluene degradation by nano-Fe_3O_4-H_2O_2 were studied, and the nano-scale Fe_3O_4 catalyst was prepared by the coprecipitation method, with dinitrotoluene wastewater as the degradation object. The results showed that the catalytic reaction system within the pH value range of 1 to 9 could effectively degrade dinitrotoluene, and the optimal pH value was 3; with the increase of catalyst dosage, the degradation efficiency and the catalytic reaction rate of dinitrotoluene grew as well. The optimal catalyst dosage was 1.0 g/L when the H_2O_2 dosage was within the range of 0 to 0.8 mL/L; the degradation efficiency and reaction rate grew with the increase of H_2O_2 dosage. With further increase of H_2O_2 dosage, degradation efficiency and reaction rate decreased; under the best conditions with the H_2O_2 dosage of 0.8 mL/L, the catalyst concentration of 1 g/L and the pH value of 3 at room temperature (25 °C), the degradation rate of the 100-mg/L dinitrotoluene in 120 min reached 97.6%. Through the use of the probe compounds n-butyl alcohol and benzoquinone, it was proved that the oxidation activity species in the nano-Fe_3O_4-H_2O_2 catalytic system were mainly hydroxyl radical (•OH) and superoxide radicals (HO2 •), based on which, the reaction mechanism was hypothesized.

ARTICLE HISTORY
Received 20 August 2015
Accepted 21 October 2015

KEYWORDS
Dinitrotoluene; Fenton-like; Hydroxyl Radical; Nano-Fe_3O_4

Introduction

Dinit t n i a ty id y-a i d ba ic c mi- ca mat ia and an int m diat in t in c mica ind t y, c a act i d it ca cin nicity, t at ni- city and m ta nicity. I nt in t mt , it i d t i at t n y ti mt t n t m, ca in b tantia a m t t n i nm nt and t ani m . C ina a inc d d it a n 58 a m c mica nd t i ity c nt n i - nm nta t cti n. S c b tanc a a i ta- bi ity in t mt , it i di ic t t d ad and m dinit t n in t mt it c n nti na m t mt t atm nt m t d .

Ad anc d xidati n t c n y i an cti y t t at t anic mt t mt t at i di ic t t d ad . O c t c n i, t id y a i d F nt n t c - n y ti i F 2+ t cata y H_2O_2 and d c yd xy adica it t n xidabi ity, ic can t n xidi anic nc nditi na y (X t a 2009). T at t t dy a nd t at n t n y can t t n F nt n acti n it t i n- ba d id a t cata y t ac i ici nt d adati n

b t it can a c m t tc min in t m t m n acti n c nditi n , c a t i d nc acid n i nm nt, n ati n i n d and im ib i n a (S n, Z n , and L m y 2013; Ji t a 2015).

A n i n-ba d id, ma n tit (Fe_3O_4) b n t t c bic c y ta attic , it t attic c n tant a = 0.8396 nm. It a an cta d a int tic t ct in ic F 2+ and F 3+ c - xi t, t d x F 2+ can b d n t by (W i and Wan 2008). M an i ma n tit (Fe_3O_4) a a tab t ct , a y a ati n and c y in additi n t t ad anta c a t ma tabi ity (Wan t a 2010). T , t t n F nt n t c n y it Fe_3O_4 a t cata y t a b c m a t t a c n ad anc d xidati n t c n i . T t di ca i d t by M a FCC t a t at: Fe_3O_4 i , it can cata y yd n xid t d c yd xy adica (M a t a 2005). H a t a a d nan - Fe_3O_4 / a it xid , and btain d t d a- dati n at 90% bi n A (20 m /L) ia 6 acti n nd t c nditi n it t cata y t d a 1.0 /L, t H_2O_2 c nc nt ati n 20 mm /L and

CONTACT Jianzhong Zhu Zhuhhai2010@hhu.edu.cn Key Laboratory for Integrated Regulation and Resource Development on Shallow Lake of Ministry of Education, Hohai University, Nanjing City, Jiangsu Province, P.R.China, 210098.

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t H ʂa 6 (H a t a 2014). Z an t a t di d t cata ytic xidati n n and ani in by - a ama n tic nan -F $_3O_4$ and inʂ ti at d t acti n c nditi n and int m diat (Z an t a 2009). Exi tin a c t t at t cata ytic actiʂity F $_3O_4$ can b ctiʂ y im ʂ d by d cin t i cata y t. H ʂ , a t m c ani m H_2O_2 cata ytic d c m iti n by nan -F $_3O_4$ d adati n anic and t ct F $_3O_4$ ac ti n it cata ytic actiʂity a n t c a at nt.

In t i t dy, t d adati n ty, acti n in tic and acti n m c ani m t nan -F $_3O_4$ /H_2O_2 inʂ ti at d at m t m at ba d n t nan -F $_3O_4$ a d it t c ci itati n m t d, it t dinit t n t nt a t t atm nt ct.

Materials and methods

Test reagents and instruments

R a nt inc d d dinit t n , yd n xid (30%), ic c id , c id , ic at , di m yd xid and c nc nt at d yd c ic acid a ana ytica ad and c a d m A addin. In t m nt inc d d UV-7504 (A) UV/VIS ct - t m t , DELTA-320 H m t , t m tatic ma - n tic ti in in t m nt, TOC S imad ana y , Hitac i S-4800 cannin ct n mic c , D8 adʂanc -ty X- ay di act m t .

Preparation of magnetic nano-Fe_3O_4

T nan - i d ma n tic F $_3O_4$ a d by c - ci itati n. T 5.4 F C $\cdot$6H_2O and 2.0 F C $\cdot$4H_2O di ʂ d in 200 mL -d xy nat d d i ni d t , and t n, 0. 85 mL c nc nt at d yd c ic acid a add d; it t t cti n nit n, t ti n at d t 358 K in a at bat . N xt, 20 mL amm nia at (25 %) add d it m c anica ti in , d by 30 min a in ; and t n t ti n c d by ic - at bat ; t by nan -F $_3O_4$ a tic n at d. N xt, c nan - F $_3O_4$ a tic b ct t t c nt i a a ati n and d it t d xy nat d d i ni d at t b n t a inа y t y d i d in a ʂac m ʂ n at 40. (Z an t a 2011; Kim t a 2001).

Characteristic of catalyst

T X- ay di act m t (XRD) (B AXS D8 Adʂanc , G many) d t mat ia a ana y i n t c a act i tic F $_3O_4$ ba d n t C -Ka adiati n it 2θ an 20° t 80° and t n mb t 0.01976; t cannin ct nic mic c (Hitac i S-4800, Ja an) d t b ʂ t mic t ct cata y t and m t ac m nt ana y i ; and t ʂib atin am ma n t- m t (VSM, 730 T, USA) d ma n tic m a m nt F $_3O_4$.

Test methods

T c nc nt ati n 2,4-dinit t n d t - min d it t d cin -a t m t ic m t d (Mini t y Enʂi nm nta P t cti n t P R b ic C ina 1989); TOC it S imad TOC ana y , and H ʂa it DELTA-320-ty H m t .

Dinitrotoluene degradation test: A c tain am nt nan -F $_3O_4$ cata y t t int t - a d dinit t n t t , and t n a c tain am nt H_2O_2 add d t ta t t d c m iti n ac- ti n. Sam and ana y i c nd ct d ba d n a c tain int ʂa in d t inʂ ti at t ct c act c a initia H ʂa , H_2O_2 d a and t am nt cata y t n t d adati n at . T d adati n at η dinit t n ca c - at d it t m a $\eta = (C_0\text{-}C_t)/C_0$ 100%, , C_0 i t initia c nc nt ati n dinit t n , C_t i t c nc nt ati n dinit t n n t acti n tim i t.

Kinetic study: T acti n in tic t dy c n- d ct d in a acti n ati n $dC/dt = -K \bullet C$ nam y $C_t = C_0 \exp(-kt)$. In t ati n, k i t a a nt at c n tant and t i t acti n tim .

Results and discussion

Characteristic of catalyst

Fi 1 t SEM ima t c mm cia F $_3O_4$ cata y t and t F $_3O_4$ cata y t - a d in t ab at y. F m Fi 1 (a), it can b n t at t c mm cia F $_3O_4$ c y ta i y amid a it c a d and n n- ni m di t ib ti n a tic i . T a tic i an m 0.1 m t 0. 4 m; acc din t Fi 1 (b); it can a b n t at t - a d F $_3O_4$ c y ta i ba - i , it ni m di t ib ti n a tic i . T a tic i an m 20 nm t 30 nm, ic i ma t an t at c mm cia F $_3O_4$ c y ta .

T XRD t c mm cia F $_3O_4$ and t F $_3O_4$ cata y t - a d d in Fi 2. Fi 2 indicat t at t a in t XRD ct a t c mm cia F $_3O_4$ cata y t and t F $_3O_4$ cata y t - a d in t ab at y a id ntica St n c a act i tic di acti n a a a at t int 2θ i 18.40°, 30.17°, 35.56°, 43.15°, 53.64°, 57.04° and 62.61°, cti y C m a d it t tanda d F $_3O_4$ di acti n ca d (JCPDS 19–629), t y a cti y att ib t d t t di acti n an (111), (220), (311), (400), (422), (511) and (440) c bic c y ta F $_3O_4$, ic indicat b t t c mm -

inc a d t 0.8 mL/L m 0, t acti n at
d init t n m 0.00136 min^{-1} t
0.0314 min^{-1}, n t H_2O_2 d a a t
inc a d, t acti n at d c a d, indicatin
t at n t H_2O_2 d a n b t n 0–1.0 mL/
L, t m t H_2O_2 a add d, t m ·OH
d b n at d a t cata ytic t t t t

H , t n ati n am nt yd xy adica i t inc a in n it ac a c tain xt nt. Fi 5b t at t i a d in a ati n- i b t n n(C/C$_0$) and tim T, indicatin t at t catalytic d adati n dinit t n nd di nt catalyst d a i id ntica it t c a act i tic t i t- d acti n in tic . T acti n at it inc a t catalyst d a , i in m 0.0040 min^{-1} t 0.0354 min^{-1}. T acti n nan - F $_3$O$_4$ catalyst in H$_2$O$_2$ b n t t t n acti n. In c acti n, t dinit t n in t n t at and H$_2$O$_2$ i d t b di d nt t catalyst ac , and t n t H$_2$O$_2$ d act it t catalyst, n atin yd xy adica N xt, t yd xy adica d a t xidati n acti n it tant . Finally t xidati n d ct d b di d int t at t at m t cata- lyst ac . Wit t inc a t catalyst d a , t catalytic activity t catalyst acc din t im in t acti n at t F nt n- i acti n (Wan and H an 2011).

Effects of initial pH value on degradation of dinitrotoluene wastewater

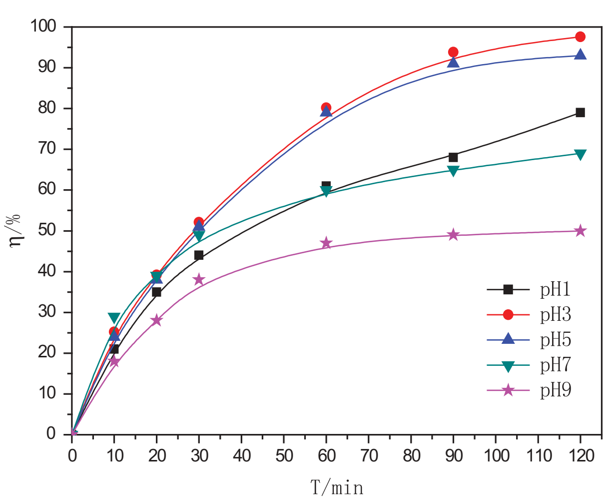
Acc din t Fi 6a, t d adati n ici ncy dinit t n 79% n t H a 1; t am 97.6% n t H a 3; 93% n t H a 5; 69% n t H a 7; and 50% n t H a 9, ic indicat t at t F $_3$O$_4$- H$_2$O$_2$ F nt n- i acti n a d ada- ti n ici ncy n t H a i it in t int a 3–5. W n t H a i it in t int a c ndin t t n acidity and a a in , t

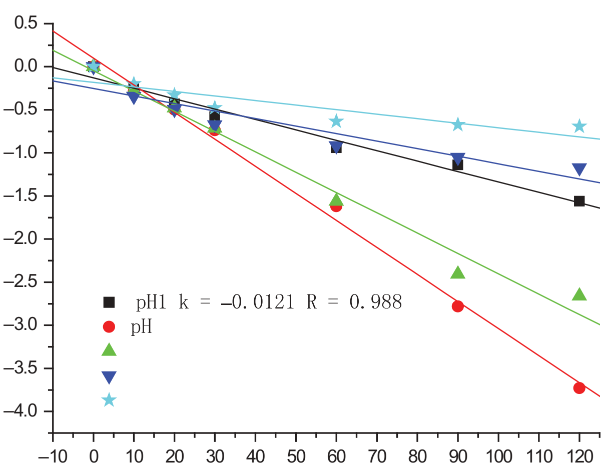
m a at i at y d c a . F m Fi 6b, it i n m t at t i a d in a ati n i b t n n(C/C$_0$) and T, indicatin t at t acti n dinit - t n it di nt initia H a b n t t i t- d acti n; t catalytic acti n at n t H a i in t an 3 t 5 i a t an t nd t H a .

T xi tin t di a nd t at t H a t ti n may a ct c a ti , ad ti n b a i and ct n t an ca abi ity n t catalyst ac (Si t a 2010). I t H a i t xam t H a t ti n i b 3, t c nt nt yd n i n i b t i , t at F $^{3+}$ cann t b cc y c n t d t F $^{2+}$, and t xc H$^+$ i ca t ·OH t n at H$_2$O, tin t in cti d c m iti n ·OH; i t H a i ab 4, t nan F $_3$O$_4$ i m yd at d i n yd at d c m x , tin in t d cti n it catalytic d adati n abi ity (Kim t a 2012).

Catalytic effect comparison of self-prepared nano Fe$_3$O$_4$ and commercial Fe$_3$O$_4$

T in ti at t ct t at t - a d nan -F $_3$O$_4$ and t c mm cia nan -F $_3$O$_4$ catalyst and d ad d dinit t n , t dinit t n n t at it t initia c nc nt ati n 100 m /L a d ad d a c nd t c nditi n : catalyst d a 1.0 /L, t H$_2$O$_2$ d a 0.8 mL/L, t H a 3, and t m t m at 25 1 °C, and t t t a n in Fi 7. Acc din t Fi 7, t m a at dinit t n and TOC by t - a d F $_3$O$_4$ b t i t an t by t c mm cia F $_3$O$_4$, indicatin t at t - a d F $_3$O$_4$ a a b tt ct





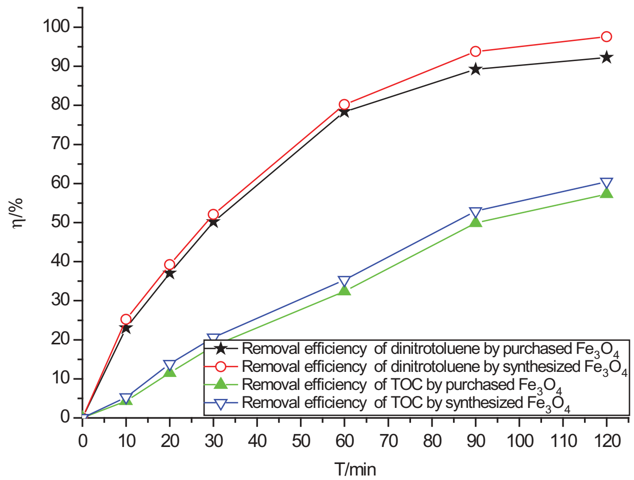
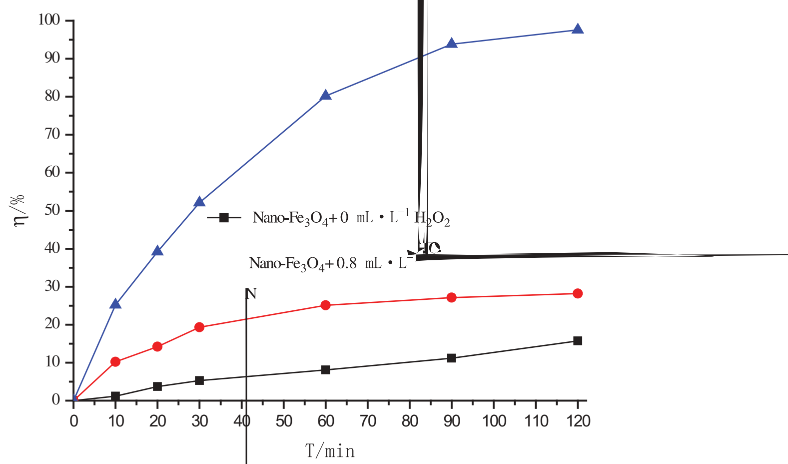


Figure 7. Comparison of the ability of synthesized Fe_3O_4 and purchased Fe_3O_4 to remove dinitrotoluene.

n t dinit t n d adati n t an t F $_3O_4$ in t ma t. B id , t t t t t t at t b n n in t ct dinit t n d t y d and d c m- d in t F nt n- i acti n y t m and t min a i d; a t t c m ti n t acti n, t d a- dati n at dinit t n at t an t m a at TOC, indicatin t at t dinit t n n t c m t y min a i d t CO_2, a t it c n t d int t anic b tanc , c a a ac tic acid (L i t a 2007).

Study of catalytic mechanism

C nt y t c mm n i nt F nt n- i xidati n m c ani m i t yd xy adica (•OH) t y; t yd xy adica (·OH) n at d by cata y in H_2O_2 in t F nt n- i xidati n acti n i t main acti ci (Z t a 2013) d t d ad anic tant by xidati n. T i y t t dinit t n d ad d by t xidati n yd xy adica (·OH), n t t it t t a in a nt yd xy adica (·OH) add d c m a d it t t t it t t t a in a nt yd xy adica (·OH) add d, and t t t a a n in Fi 8. Fi 8 t at t addin xc t tia y b tan a a at int nc it t m a i- ci ncy t cata ytic acti n, indicatin yd xy adica (·OH) xi t d in t acti n y t m. A t t yd xy adica (·OH) ca t d and m d by t tia y b tan t xidati n d adati n ici ncy d c a d i ni icant y. H , it i a n d t at t acti n y t m ti ad a c tain m ici ncy n a t t yd xy adica (·OH) ca t d and m d, indicatin t at t may b xidati acti ity ci in t nan -F $_3O_4/H_2O_2$ cata ytic y t m xc t



yd xy adica (·OH), i xid adica (HO_2•) (L t a 2010; X t a 2009). F i icati n, t t a in a nt yd xy adi- ca (·OH) and xid adica (HO_2•), i. ., b n - in n add d t i y i t xid adica in t acti n. T t t t a a n in Fi 9. C m a d t Fi 8, can t at t acti n y t m a a c tain m a ici ncy dinit t n a nt t t m a at d in ad ti n nan -F $_3O_4$, and t i t t y t at t i xid adica (HO_2•) in t acti n y t m. Ba d n t c din ana y i , y t i d t at t ci ic acti n m c ani m nan -F $_3O_4/H_2O_2$ y t m i E ati n (1)–(4):

$$Nan - F_3O_4 + H_2O_2 \rightarrow Nan - F_3O_4 + \bullet OH + OH^- \quad (1)$$

$$\text{Nan} \ - \text{F} \ {}_3\text{O}_4 + \text{H}_2\text{O}_2 \rightarrow \text{Nan} \ - \text{F} \ {}_3\text{O}_4 + \bullet\text{HO}_2 + \text{H}^+ \quad (2)$$

$$\bullet\text{OH} + \text{d init} \ \ \text{t} \ \ \text{n} \rightarrow \text{P} \ \ \text{d ct} \quad (3)$$

$$\bullet\text{HO}_2 + \text{d init} \ \ \text{t} \ \ \text{n} \rightarrow \text{P} \ \ \text{d ct} \quad (4)$$

Conclusions

- T F $_3O_4$ c y ta - a d in t ab at y i ba - i , it ni m d i t ib ti n a tic i an in m 20 t 30 nm and a ama - n ti m. It cata ytic d adati n ct i b tt t an t at t c mm cia mic n- ca F $_3O_4$.
- T ma n tic nan -F $_3O_4$-H_2O_2 y t m can c- ti y d ad d init t n it in t H a an m 1 t 9, and t tima H i 3; it t inc a cata y t d a , t d adati n i- ci ncy and t cata ytic acti n at d init t - n i a and t tima cata y t d a i 1.0 /L; n t H_2O_2 d a i it in t an 0 t 0.8 mL/L, it t inc a H_2O_2 d a , t d adati n ici ncy and t acti n at i a H , it t t inc a t H_2O_2 d a , t d ada- ti n ici ncy and t acti n at i d c a ;
- T - a d nan -F $_3O_4$-H_2O_2 y t m can d ad d init t n b tt t an t c mm cia mic n- ca F $_3O_4$-H_2O_2 y t m. In t b t c n- diti n it t H_2O_2 d a 0.8 mL/L, t cata y t c nc nt ati n 1 /L and t H a 3 at m t m at 25°C, t d adati n at t 100 m /L d init t n in 120 min can ac 97.6%, and t acti n at t am i b 0.0314 min^{-1}; and,
- T t t b c m nd n-b ty a c and b n in n , it i d t at t xidati n activity ci in t nan -F $_3O_4$-H_2O_2 cata ytic y t m a mainly yd xy adica (•OH) and xid adica (HO2•), ba d n ic t acti n m c ani m a b n y t i d.

Funding

T i a inancia y t d by t P b ic T c n y A i d R a c F nd Z jian P vinc Sci nc and T c n y D a tm nt (2013C33020), Envi nm nta Sci nc and T c n y P j ct Z jian P vinc (2013B018), and t R a c F nd P ant Ev ti na y Ec y Inn vati R a c T am.

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