

Purified terephthalic acid wastewater biodegradation and toxicity

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Abstract: The biodegradation and toxicity of the purified terephthalic acid(PTA) processing wastewater was researched at NJYZ pilot with the fusant strain Fhhh in the carrier activated sludge process(CASP). Sludge loading rate(SLR) for Fhhh to COD of the wastewater was 1.09 d^{-1} and to PTA in the wastewater was 0.29 d^{-1} . The results of bioassay at the pilot and calculation with software Ebis3 showed that the 48h-LC₅₀ (median lethal concentration) to *Daphnia magna* for the PTA concentration in the wastewater was only 1/10 of that for the chemical PTA. There were 5 kinds of benzoate pollutants and their toxicities existing in the wastewater at least. The toxicity parameter value of the pure chemical PTA cannot be used to predicate the PTA wastewater toxicity. The toxicity of the NJYZ PTA wastewater will be discussed in detail in this paper.

Keywords: fusant strain; purified terephthalic acid; wastewater; biodegradation; toxicity; software

Introduction

The pure chemical PTA has acute toxicity, sub-acute toxicity, chronic toxicity and molecular toxicity reported by (Chen, 2000; 2001; Qu, 2000; Hoshi, 1968; Chin, 1981; Xiang, 1999; Wolkowski, 1982; Yuan, 1998; Hall, 1984; Barber, 1995; Qi, 2000; 2002; Shi, 2000; Zhang, 2001; Meyer, 1984; Goncharova, 1988; Bartosiewicz, 2000; Pemmie, 2000). There was only one researcher who reported the PTA wastewater toxicity (McBee, 1987; 1988). The toxic concentrations or doses of the pure chemical PTA were as high as over 1000 mg/L or over 5% of forage amount to organisms. They might make a wrong conclusion that the PTA wastewater toxicity is too low to study. But there are at least over 5 kinds of benzoate pollutants existing in PTA wastewater including terephthalic acid(TA = PTA), phthalic acid(PA), benzoic acid(BA), 4-carboxybenzaldehyde(4-CBA) and *p*-toluic acid(*p*-tol) etc. The COD concentration of the 5 benzoates is over 70% of that of the wastewater (Kleerebezem, 1997; Cheng, 1997). So the PTA wastewater toxicity should be quite different from that of the pure chemical PTA.

This paper will report the results of the NJYZ PTA wastewater biodegradation and toxicity obtained during the pilot for 30 d. The total pilot research was conducted from November of 2003 to June of 2004 for 180 d with the functional strain Fhhh to the treatment of wastewater. Fhhh was constructed from the protoplast fusion of two fungi (*Phanerochaete chrysosporium* and *Saccharomyces cerevisiae*) and one native bacterium(*Bacillus* YZ1, isolated from NJYZ PTA wastewater treatment plant). And Fhhh got high degradability, high adaptability, high flocculation ability and generation stability from its three parental strains demonstrated in kinetics and molecular biology tests (Zhong, 2000; Yan, 2001; Tao, 2001; CARB, 2004; Chen, 2002; Zhu, 2004; Cheng, 2004).

The mathematical models were made on the relationships between the toxicity and its effective concentration for the pure chemical PTA, and between the bioassay and PTA concentration in the wastewater to *Daphnia magna* 48h-LC₅₀

(median lethal concentration) at the pilot. After the mathematical models set the environmental biotechnological informatics software3 (Ebis3) was programmed (CARB, 2003). Ebis3 was used to predicate the toxicity of NJYZ PTA wastewater. The results showed that the 48h-LC₅₀ to *Daphnia magna* for the pure chemical PTA was 10 fold of that for the PTA concentration in NJYZ PTA wastewater. It suggested that the toxically effective parameter value for the pure chemical PTA could not be used to predicate the PTA wastewater toxicity.

Biodegradation technology is the mainly direction of PTA wastewater treatment research in the world during the past 20 years. Over 70% pollutants are benzoate chemicals in PTA wastewater being the barriers to improve the biodegradation rate. The low biodegradation rate increases the treatment cost at a high level. In order to find a way to cut down the consumed energy, the anaerobic degradation technology becomes a hot point in the recent years. But the anaerobic biodegradation rate is not as high as expected. The pretreatment approaches of PTA wastewater, physics and chemistry, occurred intending to enhance the biodegradation rate (Han, 2002; Park, 2003; Crepaldi, 2002).

The results of this research will reveal the potential of Fhhh obviously in biodegradation of PTA wastewater. The sludge loading rate(SLR) i.e. the specific degradation rate (SDR) for Fhhh was 4—7 fold of that for both anaerobic strains and aerobic strains reported. And the data will suggest that the PTA wastewater toxicity is quite different from that of the pure chemical PTA. The toxicity of the NJYZ PTA wastewater will be also discussed in this paper in details.

1 Materials and methods

1.1 Starting strain and medium

Fhhh was the starting strain in this research constructed by our research group and the medium was made with the normal prescription for cultivation of fungi (Zhong, 2000; CARB, 2004).

1.2 PTA wastewater quality and process parameters

Table 1 reveals the values of NJYZ PTA wastewater quality and the process parameter values of the pilot.

Table 1 The NJYZ PTA wastewater quality and treatment process parameters at the pilot

Parameters for PTA wastewater quality	Value	Parameters for treatment process	Value
Chemical oxygen demanded(COD), g/L	3.24	Wastewater flow Q_0 , m ³ /d	6.6
Biochemical oxygen demanded(BOD ₅), g/L	2.19	Aeration influent COD S_i , g/L	1.85
Total organic carbon(TOC), g/L	1.02	Biomass in aeration tank X, g/L	1.65
Total suspended solid(TSS), g/L	0.14	Dissolved oxygen (DO), mg/L	3.8
Total nitrogen (TN), mg/L	75.30	Reaction biomass MLSS, g/L	1.63
Total phosphorus (TP), mg/L	13.95	COD in aeration tank S, g/L	0.07
Reaction temperature(T), °C	24.50	COD in effluent S_e , g/L	0.07
pH value	6.05	Sludge age(SA), d	10.29
Effective reaction volume, m ³	9.84	Hydraulic retention time(HRT), d	1.61

Notes: Each value is the mean of 30 d during the pilot; MLSS means the mixed liquor suspended solid

1.3 Measurement methods

Measurements of COD, BOD₅, MLSS, TSS and 48h-LC₅₀ to *Daphnia magna* were according to the Standard Methods(APHA USA, 1992), and TN and TP according to the methods published by China EPA(NEPAC, 2002). TOC was measured with instrument TOC-5000, USA. The 5 kinds of benzoate pollutants were analyzed with GC-MS(GC-6890, Agilent, USA).

2 Results and discussion

2.1 Results of PTA wastewater treatment at the pilot

Table 2 shows the results for PTA wastewater treatment with the functional strain Fhhh during the pilot study from May 9 of 2004 to June 10 of 2004 for 30 d. The other 4

reports were also listed in Table 2 including the monitored results of the NJYZ PTA wastewater treatment plant at the primary designation stage(Xue, 1998).

The SLR value for Fhhh was 1.09 kgCOD/(kgMLSS·d) at the pilot as shown in Table 2 being the 4—7 fold of that reported by Xue of NJYZ(Xue, 1998), Zhao(Zhao, 1994), Cheng(Cheng, 1997) and Kleerebezem(Kleerebezem, 1997). It means that the functional strain Fhhh has the potential in degradation of PTA wastewater and its degradation rate value was 4—7 fold of that for both the native anaerobic and aerobic strains reported. Using Fhhh to treat PTA wastewater could increase 445% of degradation rate, decrease 82% of the energy exhaust and save 55% of construction cost than using the native strain in A/O process in 1998.

Table 2 Comparison of the results between PTA wastewater treatment with Fhhh and the other 4 reports

Parameters	Results from NJYZ	Results from publications			
		Xue, 1998	Zhao, 1994	Cheng, 1997	Kleerebezem, 1997
Strains in reaction systems	Fhhh	Aerobic	Aerobic	Anaerobic	Anaerobic
Treatment processes	CSAP	A/O	A/O	UASB	UASB
COD in influent, g/L	3.24	2.5	1.1	4.0	3.5
PTA in influent, g/L	0.53	—	—	0.92	0.50
PTA in effluent, g/L	0.06	—	—	0.41	0.18
PTA-SLR, kgPTA/(kg·d)	0.29	—	—	0.047	0.036
Influent flow Q_0 , L/h	275	5×10^5	5	0.44	0.008
Hydraulic retention time, d	1.61	1.67	1.67	1.8	1
Effective reaction volume, L	9840	2×10^7	200	19	0.2
Running time for process, d	180	30	120	60	120
Biomass MLSS or MLVSS, kg/m ³	1.65	4.25	3.30	10.80	8.95
Sludge loading rate(SLR), d ⁻¹	1.09	0.20	0.20	0.26	0.15
Volumetric loading rate(VLR), kg/(m ³ ·d)	1.91	0.85	0.66	2.93	1.34

Notes: Each value of Fhhh is the mean of 30 d at the pilot; MLVSS = mixed liquor volatile suspended solid; A/O = anaerobic/oxygenation; UASB = upflow anaerobic sludge blanket; CSAP = carrier activated sludge process

The VLR value for Fhhh was 1.91 kgCOD/(m³·d) being lower than that for the two UASB strains as shown in Table 2. It is because that the biomass concentration MLSS of Fhhh was not as high as that of the two UASB. If the Fhhh MLSS concentration would be as high as the strains in UASB, the Fhhh VLR value would be 4—7 fold of that of the two UASB reported. The relative research results for this program showed that the Fhhh MLSS concentration could reach 6.9 kg/m³ while the value of Fhhh VLR was 7.52 kgCOD/(m³·d) being 3—11 fold of that of the other 4 strains reported listed in Table 2.

The Fhhh SLR value to PTA existing in NJYZ PTA wastewater(PTA-SLR) was 0.29 kgPTA/(kgMLSS·d)(Table 2) being 8 fold of 0.036 d⁻¹ reported by Kleerebezem(Kleerebezem, 1997) and 6 fold of 0.047 d⁻¹ reported by

Cheng(Cheng, 1997). PTA concentration in NJYZ PTA wastewater was 530 mg/L(Table 3), the ratio of PTA/COD is equal to 1/1.4(Fajardo, 1997), so 530 mgPTA/L is equal to 742 mgCOD/L. The PTA-COD concentration occupied 23% of the total COD concentration of the NJYZ PTA wastewater.

2.2 Toxicity for the pure chemical PTA and for the PTA wastewater

Table 3 shows the concentrations of the 5 benzoates in the NJYZ PTA wastewater tested with GC-MS at the pilot. The total substance concentration of the 5 benzoates was 1344.5 mg/L in which 39% was PTA of that being 530 mg/L. The total COD concentration of the 5 benzoates occupied over 70% of the wastewater(over 2268 mg/L) in which PTA-COD was 742 mg/L being 33% of 2268 mg/L.

Table 3 Five kinds of benzoate concentrations in PTA wastewater and degradation rates at the pilot

Benzoate	<i>p</i> -tol		BA		4-CBA		PA		PTA	
	Influent, mg/L	Removed, %	Influent, mg/L	Removed, %	Influent, mg/L	Removed, %	Influent, mg/L	Removed, %	Influent, mg/L	Removed, %
1	303.02	70.63	72.28	94.92	143.55	80.70	88.91	88.47	250.83	71.76
2	268.58	78.67	67.98	94.29	98.47	84.25	95.84	93.07	297.50	85.43
3	341.48	84.45	67.02	94.61	174.76	89.25	69.28	89.33	419.17	88.80
4	508.96	69.94	57.44	93.53	153.26	82.25	91.21	88.59	621.67	72.37
5	438.67	78.23	150.31	94.21	251.73	84.40	137.41	92.43	394.17	93.16
6	385.59	83.44	86.17	97.46	325.24	91.06	125.87	94.25	580.83	90.22
7	554.23	79.77	140.74	95.31	266.30	93.36	116.63	94.51	825.84	94.05
8	498.47	86.28	220.68	97.52	205.27	94.33	86.61	94.42	765.00	96.18
9	503.61	87.73	108.19	98.58	156.73	92.78	57.74	91.65	735.83	94.70
Mean	420.90	80.10	104.00	95.90	193.80	88.40	95.80	91.80	530.00	88.20

Note: Each number is the mean of 10 samples

The toxicities of the pure chemical PTA are listed in Table 4. There was only one researcher who reported the PTA wastewater toxicities also listed in Table 4 (McBee, 1987; 1988). There were 5 kinds of benzoate pollutants, PTA, PA, BA, *p*-tol and 4-CBA, at least existing in the PTA

wastewater. Actually the toxicity of PTA wastewater to organisms should be dedicated to the multiple concentration and response of the 5 benzoate compounds and not only to the PTA concentration in the wastewater.

Table 4 Toxicities of the PTA pure chemical and PTA wastewater reported

Organisms	Dose or concentration	Response parameters	References
<i>T. wisconsinense</i>	584.33 mg/L	24h-EC ₅₀	Chen, 2000
<i>T. wisconsinense</i>	573.32 mg/l.	48h-EC ₅₀	Chen, 2000
<i>Daphnia magna</i>	3165.2 mg/L	48h-LC ₅₀	Qu, 2000
<i>Daphnia magna</i>	1111.7 mg/L	96h-LC ₅₀	Qu, 2000
Grass carp larvae	3322.2 mg/L	48h-LC ₅₀	Chen, 2001
Grass carp larvae	2306.2 mg/l.	96h-LC ₅₀	Chen, 2001
SD mouse	5000—6400 mg/kg, oral	LD ₅₀	Hoshi, 1968
Female rat	> 15000 mg/kg, oral	LD ₅₀	Chin, 1981
Female SD rat larvae	5% of feed, 90 d	20% bladder calculi	Xiang, 1999
Male SD rat larvae	5% of feed, 90 d	90% bladder calculi	Xiang, 1999
Male SD rat larvae	5% of feed, 90 d	20% kidney calculi	Xiang, 1999
Female/Male SD rat	5% of feed, 90 d	10% / 40% bladder calculi	Wolkowski, 1982
<i>Daphnia magna</i>	64—256 mg/L, > 28 d	2.9%—75.3% generation rate	Yuan, 1998
<i>Daphnia magna</i>	32—256 mg/L, twice > 28 d	37.8%—78.1% generation rate	Yuan, 1998
SD rat	20 mg/kg, injection/16 d	Cholesterol abnormal	Hall, 1984
SD mouse	1.0% of feed, 90 d	Ester enzyme inhibited	Barber, 1995
SD rat	5000 mg/kg, 90 d	NAG and ALP increased	Qi, 2000
SD rat	5000 mg/kg, oral, 90 d	α ₁ -Mg protein increased	Qi, 2000
SD rat	5000 mg/kg, oral, 90 d	AST level increased	Qi, 2000
SD rat	191.16—340.72 mg/m ³ , 90 d	NAG and ALP increased	Shi, 2000
NIH + 3T3 cell	2500 mg/L, 24—72 h	Cell toxicity	Zhang, 2001
WisTar mouse	20 mg/kg, oral, 90 d	Urinary enzyme increased	Meyer, 1984
SD rat	5000 mg/kg, oral, 90 d	24% bladder cancer	Qi, 2002
SD mouse	166 mg/kg injection, 24 h	Chromosome aberrations	Goncharova, 1988
SD mouse	166 mg/kg injection, 24 h	High rate of micro nuclei	Goncharova, 1988
WisTar mouse	5 mg/(kg·d) injection	DNA construction changed	Bartosiewicz, 2000
SD mouse	166 mg/(kg·d) injection	Gene expression changed	Pemmic, 2000
Native small mammals	PTA wastewater	Chromosomal aberrations	McBee, 1987
Wild rodents	PTA wastewater	DNA damage	McBee, 1988

Notes: *T. wisconsinense* = *Tetrademus wisconsinense*; LD₅₀ = median lethal concentration; EC₅₀ = median effect concentration; NAG = N-acetyl-beta-D-glucosaminidase; ALP = alkaline phosphatase; AST = aspartate aminotransferase

The concentration of the pure chemical PTA for *Daphnia magna* 48h-LC₅₀ was 3165.2 mg/L (Table 5). But the PTA concentration in 62% of NJYZ PTA wastewater to *Daphnia magna* 48h-LC₅₀ was only 327.5 mg/L obtained from bioassay at the pilot (Table 5). The 48h-LC₅₀ of the pure chemical PTA to *Daphnia magna* was as high as that of

10 fold of the PTA in the wastewater. It suggests that it cannot use the toxicity parameter of the pure chemical PTA to indicate the PTA wastewater toxicity. The toxicities of the other 4 benzoates in PTA wastewater cannot be omitted or briefed as the PTA only.

Table 5 Concentrations for the pure chemical PTA reported and 5 benzoates in wastewater predicated, mg/L

Organisms and toxic effectives	PTA pure chemical		5 benzoates in PTA wastewater predicated			
	PTA	PTA	<i>p</i> -tol	BA	4-CBA	PA
<i>Daphnia magna</i> , 48h-LC ₅₀	3165.2	327.5	258.7	64.1	121.2	59.0
<i>Daphnia magna</i> , 96h-LC ₅₀	1111.7	114.9	90.8	22.5	42.5	20.7
<i>T. wisconsinense</i> , 24h-EC ₅₀	584.33	172.1	136.6	33.7	57.8	31.2
<i>T. wisconsinense</i> , 48h-EC ₅₀	573.32	168.9	134.1	33.1	61.8	30.6
<i>D. magna</i> 58% RA rate decreased	64—256	2.22	1.75	0.44	0.82	0.40
<i>Grass carp</i> larvae, 48h-LC ₅₀	3322.2	458.5	364.0	89.9	167.8	83.0
<i>Grass carp</i> larvae, 96h-LC ₅₀	2306.2	160.9	127.1	31.5	59.5	29.0
N II + 3T3 cell toxicity/cancer	2500	736.4	584.7	144.3	269.5	133.3
Wild rodents DNA damage	1799.4	530	420.9	104.0	193.8	95.8

Note: RA = reproduction rate

PA toxicity may be a good example to illustrate the other benzoate toxicities existing in PTA wastewater except PTA. Dimethyl phthalate (DMP) is one of PA esters being industrially important chemical, which is widely used for the manufacture of plastics as plasticizer. Mono-DMP is not bound covalently to the plastic resin and is therefore able to migrate into the environment through disposal or during use (Jonsson, 2003). Significantly high levels of phthalates and its major metabolite mono-phthalates were identified in 28 samples of 41 girls younger than 8 years of age at Puerto Rico who were the premature breast development patients. The phthalates identified have been classified as endocrine disruptors. There is a possible association between plasticizers with known estrogenic and antiandrogenic activity and the cause of premature breast development in a human female population (Colon, 2000). PA esters and other phthalate esters are suspected to cause cancer and chronic kidney inflammation. Known for endocrine-disrupting and estrogenic activity, PA ester interferes with the reproductive system and normal development of animals and humans. New studies also suggested that phthalate esters and their degradation products, the so-called mono-phthalates, play an important role in the development of allergic respiratory diseases. Phthalate esters have been listed as priority pollutants by the USA Environmental Protection Agency (USEPA, 1992).

The 100 mg/L of PA may be the LC₅₀ (median lethal concentration) or EC₅₀ (median effective concentration) to livingthings (Scholz, 2003). The concentration of PA in the wastewater was 95.80 mg/L (Table 3) near to 100 mg/L. So the toxicity of PA could not be omitted.

On the basis of 48h-LC₅₀ to *Daphnia magna* for the pure chemical PTA reported and for the PTA concentration in the 62% of the raw NJYZ PTA wastewater tested through bioassay at the pilot, the mathematical model were set for programming the environmental biotechnological informatics software3 (Ebis3). Ebis3 was used to predicate the PTA wastewater toxicity. The analysis results for the NJYZ PTA wastewater toxicity are shown in Table 5.

In Table 5, the concentration of PTA in the 62% of NJYZ PTA wastewater was 327.5 mg/L obtained from the bioassay at the pilot which was *Daphnia magna* 48h-LC₅₀. The value of 327.5 mg/L for PTA in the wastewater was also a parameter input into Ebis3 software to calculate the other toxicity parameter values.

It is clear that the 48h-LC₅₀ to *Daphnia magna* for the

PTA pure chemical is 3165.2 mg/L near the 10 fold of the PTA in the wastewater. The total concentration of *p*-tol, BA, 4-CBA, PA and PTA was 830.5 mg/L in the 62% of NJYZ PTA wastewater which was 48h-LC₅₀ to *Daphnia magna*. Meanwhile the concentration of PTA in the wastewater was only 327.5 mg/L (Table 5), which was 39% of 830.5 mg/L. The 830.5 mg/L of the 5 benzoates is also much lower than 3165.2 mg/L of the PTA pure chemical, it means that the 5 benzoate toxicity in NJYZ PTA wastewater is 3.8 fold of the pure chemical PTA.

The concentration of PTA existing in the raw NJYZ PTA wastewater was 530 mg/L as shown in Table 3. If the toxicity parameter value of the pure chemical PTA in Table 5 was used to judge the raw NJYZ PTA wastewater toxicity, the wastewater would have no acute toxicity to *D. magna* and *G. carp* larvae. It is then a wrong conclusion. The raw NJYZ PTA wastewater had 8 toxicities demonstrated by the bioassay and the calculation with Ebis3 shown in Table 5. The exciting thing is that the concentration of PTA existing in the effluent was 63 mg/L at the pilot. It means that there were 7 kinds of toxicities disappeared after treatment with the functional strain Fhhh at NJYZ in 2004. But the effluent still had the toxicity of decreasing the 58% reproduction rate of *D. magna*.

3 Conclusions

The SAR value for the functional strain Fhhh was 1.09 kgCOD/(kgMLSS·d) tested during the NJYZ PTA wastewater pilot study, which was 4—7 fold of that for both anaerobic and aerobic strains reported.

Fhhh had the degradation rate 445% higher, the construction cost decreased 55% and the energy decreased 82% comparing with those researched at NJYZ with the native *Bacillus* YZ1 during the designation time in 1998.

There were at least 5 benzoate pollutants existing in NJYZ PTA wastewater and 48h-LC₅₀ to *Daphnia magna* was 62% of the wastewater while the PTA was 327.5 mg/L being 1/10 of the pure chemical PTA.

The PTA in the effluent was 63 mg/L tested at the pilot. There were 7 kinds of toxicities removed through the treatment. Fhhh with two characteristics, high effectivity and high security, were demonstrated in this research.

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