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Tuan,

LAPORAN AKHIR SKIM GERAN PENYELIDIKAN FUNDAMENTAL (FRGS)

Tajuk Projek : Kinetic Modelling and Thermodynamic Study on Xenobiotic Organic Compounds Removal using Biosolids

No. Akaun : 203/PKIMIA/671085

Dengan hormatnya perkara di atas dirujuk.

2. Terlebih dahulu saya ucapkan ribuan terima kasih di atas satu salinan laporan akhir untuk projek penyelidikan seperti tajuk di atas.

3. Adalah dimaklumkan walaupun projek ini telah selesai, kerjasama Jabatan Bendahari dipohon untuk menguruskan penutupan akaun projek pada selewat-lewatnya **31 Disember 2011**. Tempoh ini bertujuan untuk menyelesaikan semua urusan tuntutan dan bayaran yang telah dibelanjakan di dalam tempoh projek. Walau bagaimanapun, tuan dinasihatkan supaya tidak mengeluarkan borang-borang pesanan baru di dalam tempoh ini.

4. Selanjutnya sila ambil perhatian terhadap perkara-perkara berikut sekiranya berkaitan:

- (i) Semua penerbitan harus merakamkan penghargaan kepada **Skim Geran Penyelidikan Fundamental (FRGS)** dan tuan dipohon mengemukakan satu salinan ke Pejabat ini.
- (ii) Bahagian Penyelidikan & Inovasi boleh/akan mengagihkan semula peralatan yang telah dibeli menggunakan peruntukan geran ini seandainya terdapat penyelidik lain yang memerlukan peralatan tersebut.

5. Akhir sekali, tahniah di atas usaha dan kejayaan pihak tuan dapat menyelesaikan projek ini dengan jayanya.

Sekian, terima kasih.

"BERKHIDMAT UNTUK NEGARA"
'Memastikan Kelestarian Hari Esok'

Yang menjalankan tugas,


(AMRA OTHMAN)
Penolong Pendaftar
Unit Pengurusan Geran & Kontrak

HAN, HAR, SIM

LAPORAN AKHIR SKIM GERAN PENYELIDIKAN FUNDAMENTAL (FRGS)


Tajuk Projek : Kinetic Modelling and Thermodynamic Study on Xenobiotic Organic Compounds Removal using Biosolids

No. Akaun : 203/PKIMIA/671085

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Pejabat Pelantar Penyelidikan
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} Disampaikan satu salinan laporan akhir projek untuk simpanan Perpustakaan

} Mohon kerjasama pihak puan untuk menguruskan penutupan akaun projek selewat-lewatnya pada **31 Disember 2011** dan mohon kemukakan satu salinan penyata kewangan terakhir ke Pejabat ini untuk tujuan rekod



FINAL REPORT
FUNDAMENTAL RESEARCH GRANT SCHEME (FRGS)

Laporan Akhir Skim Geran Penyelidikan Asas (FRGS) IPT
Pindaan 1/2009

A RESEARCH TITLE : Kinetic Modelling and Thermodynamic Study on Xenobiotic Organic Compounds

Tajuk Penyelidikan **Removal Using Biosolids**

PROJECT LEADER : Assoc. Prof. Dr Seng Chye Eng
Ketua Projek

PROJECT MEMBERS : 1. Prof. Lim Poh Eng,
(including GRA) 2. Dr. Amat Ngilmi Ahmad Sujari,
Ahli Projek 3. Ms. Khor Siok Moi,
4. Ms Ng Si Ling

PROJECT ACHIEVEMENT (*Prestasi Projek*)

B

ACHIEVEMENT PERCENTAGE			
Project progress according to milestones achieved up to this period	0 - 50%	51 - 75%	76 - 100%
Percentage			100 %
RESEARCH FINDINGS			
Number of articles/ manuscripts/ books	Indexed Journal	Non-Indexed Journal	
	2	-	
Paper presentations	International	National	
	-	1	
Others (Please specify)	-		
HUMAN CAPITAL DEVELOPMENT			
Human Capital	Number		Others (Please specify):
	On-going	Graduated	
PhD Student	1		
Masters Student			
Undergraduate Students	2	3	
Temporary Research Officer			
Temporary Research Assistant	2		
Total	7		

EXPENDITURE (Perbelanjaan)

C Budget Approved (Peruntukan diluluskan) : RM 96,068.62
Amount Spent (Jumlah Perbelanjaan) : RM 96,068.62
Balance (Baki) : RM 0
Percentage of Amount Spent : 100 %
(Peratusan Belanja)

ADDITIONAL RESEARCH ACTIVITIES THAT CONTRIBUTE TOWARDS DEVELOPING SOFT AND HARD SKILLS
(Aktiviti Penyelidikan Sampingan yang menyumbang kepada pembangunan kemahiran insaniah)

D

International		
Activity	Date (Month, Year)	Organizer
(e.g : Course/ Seminar/ Symposium/ Conference/ Workshop/ Site Visit)		
National		
Activity	Date (Month, Year)	Organizer
(e.g : Course/ Seminar/ Symposium/ Conference/ Workshop/ Site Visit)		

PROBLEMS / CONSTRAINTS IF ANY (Masalah/ Kekangan sekiranya ada)

E

Nil

RECOMMENDATION (Cadangan Penambahbaikan)

F

Nil

RESEARCH ABSTRACT – Not More Than 200 Words(*Abstrak Penyelidikan – Tidak Melebihi 200 patah perkataan*)

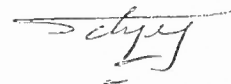
C

The objectives of this study were (1) to study the sorption of xenobiotic organic compounds onto biosolids, (2) to determine the thermodynamic parameters and isotherm constants from the equilibrium studies at different temperature, and (3) to develop or modify a kinetic model based on kinetic studies. Three non-volatile xenobiotic organic compounds, 4-chlorophenol (4CP), 2,4-dichlorophenol (DCP) and p-nitrophenol (PNP) were selected as the sorbates, whereas dried activated sludge and modified rice husk were selected as the biosorbents. Equilibrium adsorption studies were conducted by varying the operational parameters such as initial pH, contact time and initial concentrations of 4CP, DCP and PNP. The equilibrium adsorption data were fitted to the Langmuir, Freundlich, and Redlich-Peterson models to describe mathematically the sorption equilibrium and to obtain the isotherm constants. Kinetic models were developed to fit the experimental data obtained in the sorption kinetic studies involving different initial adsorbate concentrations and at selected temperatures. The thermodynamic constants of the adsorption process, ΔG° , ΔH° , and ΔS° , were determined over the temperature range of 298-318 K.

Keywords: 4-chlorophenol; 2,4-dichlorophenol; p-nitrophenol; isotherm; kinetic; thermodynamic constants

Date :
Tarikh : 09/03/10

Project Leader's Signature:
Tandatangan Ketua Projek



COMMENTS, IF ANY/ ENDORSEMENT BY RESEARCH MANAGEMENT CENTER (RMC)
(*Komen, sekiranya ada/ Pengesahan oleh Pusat Pengurusan Penyelidikan*)

H

Name:
Nama:

Signature:
Tandatangan:

Date:
Tarikh:



Bioregeneration of activated carbon and activated rice husk loaded with phenolic compounds: Kinetic modeling

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ABSTRACT

A kinetic model consisting of first-order desorption and biodegradation processes was developed to describe the bioregeneration of phenol- and *p*-nitrophenol-loaded powdered activated carbon (PAC) and pyrolyzed rice husk (PRH), respectively. Different dosages of PAC and PRH were loaded with phenol or *p*-nitrophenol by contacting with the respective phenolic compound at various concentrations. The kinetic model was used to fit the phenol or *p*-nitrophenol concentration data in the bulk solution during the bioregeneration process to determine the rate constants of desorption, k_d , and biodegradation, k . The results showed that the kinetic model fitted relatively well ($R^2 > 0.9$) to the experimental data for the phenol- and *p*-nitrophenol-loaded PAC as well as *p*-nitrophenol-loaded PRH. Comparison of the values of k_d and k shows that k is much greater than k_d . This indicates clearly that the desorption process is the rate-determining step in bioregeneration and k_d can be used to characterize the rate of bioregeneration. The trend of the variation of the k_d values with the dosages of PAC or PRH used suggests that higher rate of bioregeneration can be achieved under non-excess adsorbent dosage condition.

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1. Introduction

Bioregeneration is defined as the renewal of the adsorptive capacity of the adsorbents through microbial action (Sublette et al., 1982; de Jonge et al., 1996a). Desorption of the biodegradable compounds will lead to bioregeneration and a renewal of the adsorption capacity of the adsorbent. In the literature, two different mechanisms have been proposed to explain the bioregeneration of the adsorbent. One of the mechanisms involves the degradation of adsorbed substance occurring on the surface of the adsorbent due to the microorganisms' exoenzymatic reaction (Perrotti and Rodman, 1974). According to this hypothesis, exoenzymes excreted by the microorganism diffuse into the pores of the adsorbent and react with the sorbed substrate. The adsorption sites are renewed when the microbial products desorb to the bulk liquid due to weaker adsorbability. Xiaojian et al. (1991), however, contended that the micropores and some of the mesopores are inaccessible to exoenzymes due to the sheer size of the exoenzymes.

The other mechanism suggests that the desorption of sorbed substrate occurs as a consequence of the concentration gradient of the substrate between the adsorbent surface and the liquid phase (Speitel et al., 1989; de Jonge et al., 1996a; Sirotkin et al., 2001; Klimenko et al., 2004; Lee and Lim, 2005). As the dissolved substrate concentration in the bulk liquid is reduced by biodegra-

ation, further desorption and biodegradation are induced. In order to promote significant bioregeneration, a large decrease in the liquid phase concentration is required, especially for strongly adsorbed compounds.

Although both the mechanisms of bioregeneration suggest desorption and biodegradation processes in sequence, several bioregeneration kinetic studies (Kim et al., 1997; Ivancev-Tumbas et al., 1998; Vinitnantharat et al., 2001; Aktaş and Çeçen, 2006a, 2007) assumed that bioregeneration is a single-step process following the first-order kinetics with respect to the adsorbed substrate. This is obviously an oversimplification. The objectives of this research are (i) to conduct kinetic studies on the bioregeneration of powdered activated carbon (PAC) and pyrolyzed rice husk (PRH) loaded with phenol and *p*-nitrophenol (PNP), respectively, and (ii) to develop a kinetic model to describe the bioregeneration process. The development of this model will enable the processes of desorption and biodegradation to be discriminated quantitatively.

2. Materials and methods

2.1. Preparation of adsorbents

In this study, PAC and PRH were used as the adsorbents. The PAC (MALBON S4), a steam-activated wood-based carbon, was purchased commercially. The PRH was prepared following the procedure described in Ng et al. (2009). Both adsorbents were dried at 104 °C for 2 h and cooled in the desiccator before use.

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not, the k values increased with increasing dosages due to decreasing substrate concentration in the bulk solution (Table 2). As was described for abiotic desorption, the adsorption of phenols occurred mainly at the micropores with stronger adsorption (Hsieh and Teng, 2000). Thus, PAC at excess dosages provided higher volume of micropores, which allowed the phenol to be adsorbed strongly onto the active sites. The decreasing k_d values resulted in the lower amount of desorbed phenol and thus an increasing k values. Generally, similar trend for k values was observed for the systems involving PNP-loaded PAC and PRH (Table 3).

4.2.2. Rate constants of desorption (k_d)

For phenol- or PNP-loaded PAC prepared by contacting with the same concentration of substrate, the k_d value was observed to decrease with increasing PAC dosages (Table 2). At the non-excess PAC dosages, the PAC was saturated with phenol or PNP which was adsorbed preferably at the micropores with strong adsorption and slower diffusion rate. When the dosages of PAC or PRH increased, it is anticipated that the adsorption of phenols would be biased toward the micropores. Under the excess adsorbent condition, phenols would be predominantly adsorbed at the micropores. Therefore, under both conditions, the desorption rate constant was found to decrease with increasing dosages.

Similar trend of k_d values was observed for PNP-loaded PRH under non-excess PRH dosages (Table 3). However, under excess PRH dosages, i.e. 20–32 and 8–16 g L⁻¹ for PNP-loaded PRH prepared by contacting with 400 and 200 mg L⁻¹ PNP, respectively, the k_d values were found to be fairly constant. This can be explained by the higher percentage of micropores in the PRH, i.e. approximately 65% compared to 38% for PAC (see Ng et al., 2009). At excess dosages of PRH, the adsorption of PNP mainly occurred at micropores irrespective of the dosages of PRH used. Thus, the rate constant of desorption would be expected to be independent of the dosages of PRH used.

For phenol-loaded PRH, it was found that the model (Eq. (9)) did not fit well to the experimental data (Fig. 3a and b). The model could not account for the convex shape (the 'hump') of the observed phenol concentration which appeared at the later stage of the bioregeneration process. The convex shape of the time course of phenol concentration could be explained by postulating that there was in fact two rates of desorption with the fast desorption of phenol from large pores and external surface of PRH and the slower desorption from micropores at a slower rate but in larger amount. Consequently, the amount of desorbed phenol was readily degraded at the initial stage of the time courses of phenol concentrations in the bulk solutions as shown in Fig. 3b and of the time course of phenol concentration at 5.0 g L⁻¹ of phenol-loaded PRH as shown in Fig. 3a. However, much larger amount of phenol desorbed from the micropores at the later stage might have exerted an inhibitory effect on the biodegradation rate resulting in a build-up of phenol concentration thus the appearance of the 'hump'.

In more quantitative terms, the relationship between the rate constants of desorption from the micropores ($k_{d,1}$) and non-micropores ($k_{d,2}$) with the generated rate constant of desorption (k_d) could be expressed as follows:

$$k_d = \frac{k_{d,1}q_1 + k_{d,2}q_2}{q_1 + q_2} \quad (10)$$

where q_1 and q_2 are the amount of adsorbed phenol (mg g⁻¹) at the micropores and non-micropores, respectively, whereas $k_{d,1}q_1$ and $k_{d,2}q_2$ are the rates of desorption from the micropores and non-micropores, respectively. For phenol-loaded PAC and PNP-loaded PAC/PRH, the relatively good fit of the proposed model (Eq. (9)) to the experimental data of residual substrate concentrations could be explained by the premise that the values of $k_{d,1}$ and $k_{d,2}$ were

comparable in those cases and thus a single-value of k_d could be generated at each adsorbent dosage. In the case of phenol-loaded PRH, the assumption of a single-value k_d is not valid as the values $k_{d,1}$ and $k_{d,2}$ would be different.

By comparing the values of the rate constant of desorption obtained under the abiotic (Table 1) and biotic conditions for PNP-loaded PAC (Table 2) and PNP-loaded PRH (Table 3) prepared by contacting with 400 mg L⁻¹ PNP, the rate constants were found to be larger under biotic condition. In the presence of PNP-adapted biomass, the concentration of desorbed PNP in the bulk solution was reduced by biodegradation. Thus, higher concentration gradient between the substrate on the adsorbent and in the bulk solution induced further desorption. The percentage of increase in the values of the rate constant was found to be greater under excess dosages of PNP-loaded PAC and PRH. This can be explained by the increasingly higher values of the rate constant of biodegradation, k , when excess dosages of PAC or PRH were used.

4.2.3. Rate of bioregeneration

Comparison of the values of k_d and k shows very clearly that k is much greater than k_d indicating that desorption is the rate-determining step for bioregeneration. This was also observed by Aktaş and Çeçen (2006a). Thus, k_d can be used to characterize the rate of bioregeneration. In addition, it was observed that the value of k_d was dependent on the dosage of the adsorbent used and was greater under non-excess adsorbent dosage condition.

5. Conclusions

The proposed kinetic model for bioregeneration which consists of first-order desorption and biodegradation processes fitted relatively well ($R^2 > 0.9$) to the experimental data on the change of the residual phenol or PNP concentration with time in the bulk solution for phenol- and PNP-loaded PAC as well as PNP-loaded PRH. The results showed that the biodegradation rate constant, k , was much greater than the desorption rate constant, k_d , indicating that desorption is the rate-determining step of the bioregeneration process. The trend of the dependence of the k_d values on the dosages of PAC or PRH used suggests that higher rate of bioregeneration can be achieved under non-excess adsorbent dosage condition.

Acknowledgement

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Treatment of *p*-nitrophenol in an adsorbent-supplemented sequencing batch reactor

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The objective of this research was to evaluate the treatment of *p*-nitrophenol (PNP) as a sole organic carbon source using a sequencing batch reactor (SBR) with the addition of adsorbent. Two types of adsorbents, namely powdered activated carbon (PAC) and pyrolysed rice husk (PRH) were used in this study. Two identical SBRs, each with a working volume of 10 L, were operated with fill, react, settle, draw and idle periods in the ratio of 2:8:1:0.75:0.25 for a cycle time of 12 h. The results showed that, without the addition of adsorbent, increasing the influent PNP concentration to 200 mg/L resulted in the deterioration of chemical oxygen demand (COD) removal efficiency and PNP removal efficiency in the SBRs. Improvement in the performance of the SBR was observed with the addition of PAC. When the dosage of 1.0 g PAC/cycle was applied, COD removal of 95% and almost complete removal of PNP were achieved at the influent PNP concentration of 300 mg/L. The kinetic study showed that the rates of COD and PNP removal can be described by the first-order kinetics. The enhancement of performance in the PAC-supplemented SBR was postulated to be due to the initial adsorption of PNP by the freshly added and the bioregenerated PAC, thus reducing the inhibition on the microorganisms. The PRH was found to be ineffective because of its relatively low adsorption capacity for PNP, compared with that of PAC.

Keywords: sequencing batch reactor; *p*-nitrophenol; treatment; adsorbents

Introduction

The presence of phenolic compounds in the environment is a cause of major concern because of their toxic effects on human health. *p*-Nitrophenol (PNP) is one of those hazardous chemicals, which is widely used in industry. It is an important intermediate in the manufacturing of azo dyes and a number of pesticides. Thus, removal of these compounds prior to discharge into the environment is needed. The removal of PNP in wastewater can be achieved by physical, chemical or biological treatment or a combination of these treatment processes [1–5]. The most widely used method for the removal of PNP is biological treatment [6–14].

The application of simultaneous adsorption and biodegradation processes in the same reactor is known to be effective for the removal of both biodegradable and non-biodegradable contaminants, including toxic compounds, in various kinds of wastewater. Previous studies have shown that the combination of powdered activated carbon (PAC) and biomass in the same aeration basin of a conventional continuous-flow activated sludge system, known as the PACT process, was effective in the removal of toxic pollutants present in various types of wastewater [15–17]. The sequencing batch

reactor (SBR) process, which carries out functions of equalization, treatment and sedimentation in the same reactor, has been recognized as a viable alternative to the conventional continuous-flow process. The SBR with PAC addition has been proven effective in treating metal-containing and dye-containing wastewaters [18–21]. However, with regards to the treatment of biorefractory compounds such as PNP, studies [22,23] have shown that complete removal of PNP up to a loading rate of 0.6 kg/(m³·d) could be achieved with the acclimatized biomass in the SBR process without the use of adsorbent, as the SBR offers dynamic conditions which favour the induction of the synthesis of specific enzymes able to metabolize the compound.

The addition of adsorbent to an activated sludge process is only warranted if there is a significant improvement in the treatment, as the disposal of the additional quantities of generated sludge poses an environmental problem. To date, reports on the treatment of biorefractory compounds in SBRs supplemented with adsorbent are lacking. Therefore, the objective of this research was to evaluate the treatment of PNP under SBR operation with the addition of adsorbent. Two types of adsorbents, namely PAC and pyrolysed rice husk (PRH), were used in this study.

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Table 3. Pseudo first-order rate constants for COD removal, k_{COD} (/min) and for PNP removal, k_{PNP} (/min) for various operational phases in reactor RP.

Phase	$k_{\text{COD}} \times 10^2$	R^2	$k_{\text{PNP}} \times 10^3$	R^2
I	1.0	0.956	1.0	0.934
II	1.5	0.819	1.1	0.965
III	1.9	0.829	1.1	0.922
IV	7.1	0.915	3.0	0.827
V	3.5	0.842	15.1	0.914
VI	3.2	0.702	18.1	0.972
VII	0.5	0.825	–	–
VIII	0.8	0.724	–	–

Acknowledgements

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