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Integrated treatment scheme of a biofilter preceded by a two-bed cyclic adsorption unit treating dynamic toluene loading

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Abstract

Biofiltration for volatile organic compound (VOC) control in waste gas streams is best operated at steady contaminants loading. Variations in contaminant loading are, however, common in real applications. In order to provide long-term stable operation of a biofilter under adverse contaminant feeding conditions, a dual-fixed bed adsorption system involving a two-step cycle, i.e., adsorption and desorption, was proposed in this study. The beds of the adsorption unit were packed with granular activated carbon, and the unit was installed in-series before the biofilter. At transient toluene loadings averaging 46.9, 56.3, and 65.9 g/(m³ h), a biofilter (total bed volume of 2.72×10^{-3} m³) with pre-cyclic adsorber/desorber unit (total volume of 2.06×10^{-4} m³) successfully reduced the waste gas toluene concentration below 5 mg/m³, while similar behavior was not achieved by a stand-alone biofilter (total volume of 2.72×10^{-3} m³). The net effect of the two-bed adsorption unit was to attenuate the peaks of toluene concentration, and to make the VOC-laden air stream amenable for biofiltration. It was concluded in this study that the integrated treatment scheme compared to a stand-alone biofilter attained the goal of reliably treating fluctuating toluene loading with high removal efficiency.

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1. Introduction

Biofiltration has been recognized as a cost-effective technology for the purification of air contaminated with low concentrations of biologically degradable organic compounds [1-3]. A stable biofilter performance strongly relies on constant contaminant loading [4]. In practice, most off-gases contaminated with volatile organic compounds (VOCs) have variable flow rates, a wide range of contaminant concentrations, and transient loadings which limit the handling efficiency of biofiltration [5-10]. Attenuation of input fluctuation is significant for regulatory compliance, especially for sources where contaminant fluctuations are commonly encountered.

It has been reported that the use of "treatment trains" or "coupled systems" may be more advantageous when compared with any single biological technology [11]. Specifically, a biofilter preceded by a carbon filter provides an effective solution for VOC emissions control with unstable pollutant loads [12–16]. Most studies installed a single adsorber bed as a buffer unit for dampening fluctuations in contaminant loading. Weber and Hartmans [12] observed that the use of activated carbon as a buffer unit minimized fluctuation in toluene loading, and subsequently the overall removal efficiency of the combined system was improved. One recent study by Li and Moe [16] also employed a single activated carbon bed for equalizing discontinuous loading (e.g., 8 h loading/day) of acetone and toluene mixture. Although these efforts have verified the effectiveness of adsorption units in dampening biofilter performance fluctuations during transient VOC loadings, applications have been restricted with regard to the desired contaminant concentration, and the frequency and duration of contaminant loading.

In practice, when larger contaminant loading fluctuations are encountered, the buffer capacity of a single carbon adsorption bed will be quickly exhausted. Furthermore, since contaminants are mostly captured in the adsorption column before the biofilter, the biofilter will be exposed to the substrate starvation condition until breakthrough occurs in the adsorption column. Such substrate starvation significantly influences the biological activity

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in the biofilter [4,8]. Longer starvation induces a longer biofilter performance acclimation [17]. Further attention needs to be undertaken towards the buffer unit design for successful utilization of the integrated adsorption and biofilter system.

For this purpose, we proposed a dual-fixed bed adsorption unit, within which a desorption step is incorporated. The two-bed adsorption system was designed to involve a short-term cycle of adsorption and desorption, occurring in a fixed bed of adsorbent, using contaminant pressure variation as the principal operating parameter. Fig. 1 shows a typical two-bed adsorption unit with the two-step cycle employed in this study. Each step includes the feeding phase (adsorption) and the purging phase (desorption). The purging phase functions as regeneration. The net effect of the two-bed adsorption unit is to flatten and reduce the VOC concentration in the treated air which makes it amenable for biofiltration.

In this study, adsorption and desorption profiles in the twobed adsorption unit were first explored using a mathematical model to simulate performance and to optimize design parameters. The experimental plan was then designed to evaluate the performance of the two-bed adsorption unit under dynamic contaminant loadings, and to investigate the overall performance of an integrated process scheme consisting of a two-bed adsorption unit followed by a biofilter as compared to a stand-alone biofilter system.



Fig. 1. Two-bed adsorption concept.

2. Materials and methods

The experimental work was performed on two lab-scale reactors for controlling toluene as a single contaminant. One system consisted of a two-bed adsorption unit followed by a biofilter



Fig. 2. Schematic diagram of the experimental setup.

(hereafter named as the integrated unit). The other system was the control unit, in which only a biofilter was involved. The systems were maintained at a constant operating temperature of $20 \,^{\circ}$ C in a constant temperature chamber. The experimental setup is shown in Fig. 2.

The air supplied to the system was purified with complete removal of water, oil, carbon dioxide, VOCs, and particles by Balston FTIR purge gas generator (Parker Hannifin Corporation, Tewksbury, MA). The air flow to each system was set up at the rate of 2.22 L/min, regulated by a mass flow controller (MKS Model 247C four-channel read-out mass flow controller, Andover, MA). Liquid toluene was injected via syringe pumps (Harvard Apparatus, model NP-70-2208, Holliston, MA) into the air stream where it vaporized, and entered the equalizing vessel before it is evenly divided then admitted into the control unit and the integrated unit, respectively.

2.1. Adsorption unit

The system was designed for operation in a two-step cycle, i.e., simultaneously feeding (adsorption) and purging (desorption) within two identical adsorption beds. The employed EBRT for the two-bed adsorption unit was designed to be 5.6 s (at an air flow rate of 2.22 L/min). The total volume of the two cylindrical adsorption beds was 2.06×10^{-4} m³. Each bed was constructed of stainless steel with an external diameter of 2.54 cm and a length of 20.3 cm. The beds were packed with 165 g of bituminous based BPL activated carbon (Calgon Carbon Co., apparent density = 0.85 g/mL). The Freundlich adsorption isotherm parameters for toluene adsorption on the selected activated carbon were experimentally determined (*K* = 339.2 ((mg/g)/(L/mg)^{1/n}) and 1/*n* = 0.314) by using a constant volume approach method [18].

Cyclic operation of adsorption and desorption was achieved by switching the flow direction within the adsorption unit, which was generated through an electrically operated 4-way solenoid valve (ASCO 8342G 701, Florham Park, NJ) controlled by an electronic timer (Digi 42A-120; GRASSLIN Controls Corp., Mahwah, NJ). The cycle duration was set at 8 h which provided each bed to have 4 h feeding and 4 h purging. Sampling ports were installed for both the feed and exhaust gases. An additional air valve was installed to introduce supplemental fresh air within

| Table 1 | | |
|-----------------------------------|------|----------|
| Toluene square wave inlet feeding | 2 CO | nditions |

the two adsorption beds for reducing contaminant partial pressure in the other fixed bed where desorption occurs, if necessary.

2.2. Biofilter unit

Two independent parallel trickle bed air biofilters (TBAB) were employed. The biofilters were constructed of cylindrical glass sections with an internal diameter of 76 mm and a total length of 130 cm. Each section was equipped with a sampling port that extends to the center of the column. Each biofilter was packed with pelletized diatomaceous earth biological support media (Celite[®] 6 mm R-635 Bio-Catalyst Carrier; Celite Corp., Lompoc, CA) to a depth of about 60 cm. The pellets were made from sintered diatomaceous earth and are therefore principally silica (SiO₂). Their physical properties were demonstrated in a previous study [19] performed at the University of Cincinnati. The biofilters were seeded with an aerobic microbial culture preacclimating to toluene, which had been obtained from previous operation of the biofilter [20]. The biofilters were operated with co-current gas and liquid downward flow mode.

The EBRT of each biofilter employed was 1.23 min (air flowrate 2.22 L/min). Single pass buffered nutrient solution was intermittently sprayed as a fine mist onto the top of the bed media through a spray nozzle (nutrient flow-rate 1.8 L/day). The buffered nutrient solution was deionized and activated carbon filtered water, which contained all necessary macronutrients, micronutrients, and buffer as described elsewhere [21].

Since biofilter performance decreased substantially due to accumulation of excess biomass within the bed media, in situ upflow backwashing was coordinated as the biomass control strategy for removing excess biomass in the biofilter. Each biofilter was backwashed at a rate of 1 h once a week. The details of the backwashing methodology are found in our previous studies [4,19].

2.3. Toluene feeding condition

Different types of inlet concentration square waves change were considered to simulate transient emissions in the chemical industry (see Table 1). Two syringe pumps were used for this purpose. The syringe pumps were controlled by an electronic timer (Digi 42A-120; GRASSLIN Controls Corp.).

| 1 | | | | |
|---|------------|------------|------------|----------------------------|
| | Type A | Type B | Type C | Type D |
| Average loading rate ^a $(g/(m^3 h))$ | 46.9 | 46.9 | 56.3 | 65.9 |
| Frequency of square wave per hour | 1 | 1 | 1 | 2 |
| Inlet concentration | | | | |
| Peak concentration (ppmv) | 400 | 700 | 400 | 600, 400 |
| Duration (min/h) | 15 | 6 | 30 | 15, 15 |
| Base concentration (ppmv) | 200 | 200 | 200 | 200 |
| Mode of feeding | Continuous | Continuous | Continuous | Discontinuous ^b |
| | | | | |

^a For Types A and B, the average loading rate applied in the present study was subjected to the critical inlet loading $(46.9 \text{ g/}(\text{m}^3 \text{ h}))$ determined in a previous study [24]. Correspondingly, the critical inlet concentration to the toluene biofilter was 250 ppmv at 2.22 L/min air flow. Types C and D are designed to have higher loading rates compared with the critical loading.

^b Toluene loading interval = 10 h/day.

2.4. Analytical methods

Gas phase samples for toluene analysis and CO_2 analysis were measured by using a gas chromatograph (HP 5890, Series II, Hewlett Packard) equipped with a flame ionization detector (FID) and a thermal conductivity detector (TCD), respectively. Method detection limit for toluene and CO_2 were 0.5 and 300 ppmv, respectively. Liquid phase samples were analyzed for total carbon (TC), inorganic carbon (IC), and volatile suspended solid (VSS) concentration. TC and IC were determined by using a Shimadzu TOC 5000 analyzer (Shimadzu Corp., Tokyo, Japan). The VSS concentrations in the effluent and backwashing solution were determined according to Standard Methods 2540 G [22]. Details about analytical methods used are found elsewhere [4,20].

2.5. Model simulation

A mathematical model was formulated for a packed bed for simulation of the proposed cyclic operation, which consists of a toluene material balance. The linear driving force (LDF) model was incorporated into the model in order to represent the mass transfer resistance between the adsorbent and the bulk gas phase (see supplementary material for details).

For comparison, non-cyclic operation of the two-bed adsorption unit, i.e., conducted as if there were one adsorption bed, was also simulated. The simulation was performed by using plug flow homogeneous surface diffusion model (PFHSDM) which is embedded in an Adsorption Design Software (AdDesignSTM) [23]. Type 'A' feeding condition was considered in the model simulation. A summary of model input parameters is presented in the supplementary material.

3. Results and discussion

3.1. Model simulation in the two-bed adsorption unit

Fig. 3(a) provides the data collected for both the adsorption and desorption cycles together with the mathematical model simulation for cyclic operation of the two-bed adsorption unit as compared to non-cyclic operation in Fig. 3(b). It is seen from Fig. 3(a and b) that the experimental data agreed reasonably with the simulated data. In both cases, a certain amount of fluctuation in the effluent concentration was unavoidable. However, for cyclic operation, the difference in the effluent gradually decreased with time and the concentration approached 250 ppmv. It is worthwhile to note that our previous study [24] found that the inlet toluene concentration over 250 ppmv led to substantial deterioration of the biofilter performance, which defined 250 ppmv to be the critical inlet concentration. For noncyclic operation the effluent concentration was varying between 231 ± 2 and 268 ± 4 ppmv after breakthrough. This variation is less amenable to effective biodegradation in the biofilter since the inlet concentration to the biofilter exceeded the critical value (250 ppmv). It is, therefore, demonstrated that the effluent from cyclic operation was better attenuated below the critical inlet concentration (250 ppmv) to the biofilter. Furthermore, it is seen



Fig. 3. Desorption profiles of two-bed adsorption (feeding condition: Type A): (a) cyclic operation; (b) non-cyclic operation, i.e., conducted as if they were one adsorption bed. For non-cyclic operation, toluene in the effluent was detected after breakthrough has occurred, i.e., after about 400 h (the inset is a blow up after about 400 h). Simulated data (solid line) were compared to experimentally observed data (\bigcirc). The dotted line indicates the critical inlet concentration (250 ppmv) to the biofilter at 1.23 min EBRT, which was determined in a previous study [24].

that, for non-cyclic operation, more than 17 days were required before any breakthrough occurred, which will provide a long non-use period for the biofilter and might highly impact the retention of microbial activities.

In order to identify the effluent response to the duration of cyclic operation, desorption rate data were collected from one adsorber bed with flow switching (see Fig. 4). The feed flow for time, $T_{\rm f}$, was upward and the purge flow for time, $T_{\rm p}$, was downward. The data were collected for $T_{\rm f}$ at 1, 2, 4, and 6 h, and $T_{\rm p}$ was varied in such a way as to provide a ratio of $T_{\rm p}/T_{\rm f}$ that ranged from 0.03 to 1.0. Fig. 4 indicates that the desorption rate apparently decreased as the purging time increased regardless



Fig. 4. Profiles of the average desorption rate (R_D) from a single bed of the adsorber undergoing alternating loading and desorption. The feed flow for time, T_f , was upward and the purge flow for time, T_p , was downward. An R^2 value of 0.999 (P < 0.017) was obtained for the regression curve; toluene feeding concentration was 250 ppmv at 2.22 L/min air flow.



Fig. 5. Overall toluene removal performance for Type 'A' feeding condition.

of the different feeding times used. Furthermore, the desorption rate (R_D , mg/min) can be empirically expressed in terms of the relative purge/feed time (R_t) as follows:

$$R_{\rm D} = 1.538 + 0.468 \,\mathrm{e}^{-2.892R_t} \tag{1}$$

Also R_D can be written as $d(M_D)/d(T_p)$, where M_D is the net mass desorbed during T_p . Integration of Eq. (1) gives:

$$M_{\rm D} = 1.538T_{\rm p} - 0.162T_{\rm f}({\rm e}^{-2.892T_{\rm p}/T_{\rm f}} - 1)$$
(2)

where M_D is the net mass desorbed. It is worthwhile to note that the above equation is only valid when the adsorber bed is not fully exhausted. If the feed and purge times are identical then the net mass desorbed during the purging process is given by:

$$M_{\rm D} = 1.691 T_{\rm p}$$
 (3)

Hence, R_D is 1.691 mg/min, demonstrating that the desorption rate is independent of the cycle duration if the purge and feed times were identical.

3.2. Toluene removal performance in the integrated system

The integrated unit and a stand-alone biofilter serving as a control unit were initially operated under a square wave Type 'A' feeding condition (see Table 1) for 48 days. The overall toluene removal efficiency and effluent concentration for the two units are provided in Fig. 5 as logarithmic plots in order to compare them over a wide range of time. It is seen from Fig. 5 that the integrated unit did not require any initial acclimation period for maintaining over 99% removal while the control unit required more than 10 days (240 h). It is believed that the inlet concentration exceeded the capacity of the biofilm present in the control unit. Therefore, the microbial population present needed to be acclimated to the higher concentration of toluene, i.e., to build up sufficient biomass. In case of the integrated unit, the adsorption unit reduced the peak concentrations being received by the biofilter. This observation demonstrated the ability of the two-bed adsorption unit to serve as a pulse-dampening unit during initial acclimation of the biofilter. This goal was achieved by abating toluene release from the adsorption unit. However, it



Fig. 6. Effluent performance on Day 42 for Type 'A' feeding condition. The dotted line indicates the square wave changes for the inlet concentration; effluent concentrations in the control unit (\bullet) and in the integrated unit (\bigcirc). The arrows indicate changes of air direction for cyclic operation in the two-bed adsorption.

is worthwhile to note that the integrated unit provided temporary drops to approximately 92% removal efficiency after day 2. One possible explanation for this is that the base concentration exiting the adsorption unit increased suddenly after 12 h operation (see Fig. 3(a)), which induced a load fluctuation in the biofilter.

After the initial 10 days of operation, the effluent concentrations from the integrated unit ranged from the detection limit of 0.5 ppmv to approximately 5 ppmv and the corresponding removal efficiencies were comparatively stable at the 99% level with some temporary drops to about 97.5%. The temporary drops in removal efficiency were mainly caused during acclimation after periodic media backwashing for biomass control. Immediately after backwashing, toluene removal efficiency dropped below 80%, and then it recovered to the 99% level in less than 10 h. In contrast, the control unit showed large fluctuations in the effluent during the same operational period. Frequent drops in removal efficiency below 90% were observed consequent to the peak square wave concentration (400 ppmv) of toluene feeding.

Fig. 6 shows a typical daily performance of both systems under a square wave Type 'A' feeding condition. It is clearly seen from Fig. 6 that the effluent performance of the integrated unit was apparently independent of the feed toluene loading. However, subsequent to cyclic operation in the two-bed adsorption unit, temporary peaks of the effluent were seen with concentration below 5 ppmv. On the other hand, the control unit provided effluent performance strongly contingent on the feeding wave concentration.

3.3. Carbon balance

The cumulative carbon of toluene consumed (C_{inlet}) in both systems (integrated and control unit) was compared to the cumulative carbon produced (C_{outlet}) within the biofilter during the experimental period for the feeding square wave condition Type 'A'. It is seen from Fig. 7 that the carbon recovery in the control unit was 97.4%, which is good, within the experimental accuracy, while the carbon recovery in the integrated unit was 81.4%. This could indicate that the difference between the integrated unit and the control unit resulted from the accumulation of



Fig. 7. Carbon recovery for Type 'A' feeding condition. The cumulative carbon produced (C_{outlet}) is estimated as the net analysis of effluent carbon in the gas and liquid streams coupled with the VSS loss from the biofilter by assuming that a typical cellular composition for a heterogeneous microorganism can be represented by $C_5H_7O_2N$.

toluene in the adsorption beds, which is estimated to be 23.525 g toluene (11.17 mol C of inlet toluene for 48 days \times 16%, the difference in carbon recovery between the two units). The theoretical toluene adsorption capacity on BPL activated carbon used in the present study is 0.335 g toluene/g activated carbon [18]. This was estimated from the peak feed concentration. Thus, the toluene holding capacity in the two-bed adsorption unit with 165 g adsorbent is 55.275 g toluene. It is, therefore, interesting to note that the adsorption beds were adsorbing 42.6% of the theoretical toluene holding capacity during the experimental period. On the other hand, the accumulation of toluene in the adsorption beds based on Eq. (3) is 30.15 g toluene for the experimental period, which indicates that the adsorbed toluene was 54.5% of the theoretical toluene holding capacity. The difference of percentile saturation between the two estimates is reasonably good, and within the experimental accuracy. The fact that the empirical loading was less than the peak loading is lower is expected since the average feed concentration is lower than the peak square wave concentration.

3.4. Effluent response after the restart-up following non-use periods

When the buffering capacity of the adsorption system is exhausted, regeneration of the adsorbents needs to be undertaken. In the present study, further regeneration could be achieved by using the supplemental air valve installed within the adsorption unit (see Fig. 2). This allows further reduction in gas partial pressure of the desorbing adsorbent bed. An increase in the running cost of the system is however unavoidable for this purpose. However, it is worthwhile to note that repeated periods of non-use, such as weekend and holidays are common in most chemical industries. These non-use periods can be considered as a method for regeneration whereby the adsorption system can be used only for desorbing the contaminants to the biofilter. Considering Eq. (2) (if $T_f = 4$ h and $T_p = 2$ days), a 2-day period of non-use allows 8.1% of the theoretical toluene holding



Fig. 8. Effluent performance after non-use period for Type 'A' feeding condition. During non-use periods pure air passed through the system.

capacity within the beds to be released. Thus, the integrated unit can be assigned for treating waste air from discontinuous processes that generate transient emissions, together with weekend recess. This would effectively retain the buffer capacity of the adsorbent unit when the process industry is back to normal shift hours. Furthermore, it should be noted that the biofilter recovery after non-use is one of the important factors for assessing biofilter performance. It is interesting to notice from Fig. 8 that after non-use periods, the integrated unit had superior reacclimation performance compared with the control unit. There was no apparent reacclimation time in the integrated system because the biological activity in the biofilter had been supported by incessant substrate release from the adsorption system. On the other hand, the control unit was exposed to substrate starvation condition whereby longer reacclimation was encountered.

3.5. Further evaluation

Having demonstrated the effectiveness of the integrated unit for Type 'A' square wave (see Table 1), its operation was further extended to different feeding conditions (see Table 1)—higher



Fig. 9. Effluent response and biological activity with respect to different feeding conditions. (a) Eight hours average effluent concentration: the box plot provides the 8 h average effluent concentration across the range of experimental periods, stretching from the lower hinge (defined as the 5th percentile) to the upper hinge (the 95th percentile). (b) First order reaction rate constant (an estimate of biological activity): the data presented represent the average values for peak loadings (\bigcirc , integrated unit; \bullet , control unit) and base loading (\triangledown , integrated unit; \blacktriangle , control unit) at each feeding condition.

peak concentration (Type B); more frequent peak concentration (Type C); higher and more frequent peak concentration (Type D). The effluent responses to different feeding conditions are summarized in Fig. 9(a). Interestingly, it was observed for Type 'D' square wave which was operated for 10 h/day no acclimation periods were apparently observed in the integrated unit, while about 2 h of acclimation in the control unit was perceived after 14h starvation each day (data is not presented). Hence, the comparison provided in Fig. 9(a) was based on using an 8 h time weighted average (TWA) effluent. If one considers toluene exposure to the occupational environment, it had been noted that the majority of people suffered acutely from mucous membrane irritation beyond a level of 5 mg VOC/m³ [25]. Thus, an exposure limit of 5 mg VOC/m³ had been adopted by the American Industrial Hygiene Association (AIHA). It is seen from Fig. 9(a) that the integrated unit successfully reduced the waste gas toluene concentration below this level (5 mg/m^3) for all cases. Similar behavior was not achieved by the control unit over any experimental period. The 8 h TWA of the effluent from the control unit was 34.7 ± 15.7 , 31.7 ± 16.4 , 10.9 ± 8.4 , and 116.2 ± 54.4 ppmv for the feeding square wave conditions of Types A–D, respectively.

In order to determine differences in biological activity of each unit, first order kinetic constants was obtained as an estimate of biological activity. The following equation [26] was used to model the kinetics:

$$\frac{C_{\rm e}}{C_{\rm i}} = \exp\left(-\frac{LK_{\rm 1}}{mU_{\rm a}}\right) \tag{4}$$

where C_e and C_i are the effluent and influent biofilter toluene concentrations, *L* the biofilter media height (60 cm), K_1 the first order reaction rate constant (an estimate of microbial activity), *m* the distribution coefficient for the VOC between liquid and gas phases which is 0.280, dimensionless Henry's law constant, and U_a is the superficial velocity (0.00816 m/s). As seen in Fig. 9(b), estimates of microbial activity were calculated for peak loading and base loading at each feeding condition. It is seen from Fig. 9(b) that more vigorous and stable biological activities were achieved by the integrated unit. It is further seen from Fig. 9 that the biological activities in the control unit deteriorated as the degree of load fluctuation increased. This supports the contention that any change in the substrate flux strongly affects the biological activity [4,8].

4. Conclusion

As a final engineering observation, the results of the present study are particularly true when waste gas emissions appear to be similar to the different square wave options explored in the present study. The present study demonstrated that the net effect of the two-bed adsorption was VOC concentration stabilization that made it amenable for effective stable biodegradation. The two-step cycle of adsorption and desorption in the two-bed adsorption unit successfully performed three particular functions: a polishing unit to abate the initial acclimation of the biofilter; a buffering unit to ameliorate the biofilter performance during load fluctuation; a feeding source for the biofilter without any feeding phase when the biofilter is exposed to non-use periods such as shut down during weekends and holidays.

Once the VOC concentrations in the waste gas stream are known, model simulation will aid the design of the two-bed adsorption unit proposed in this study, and will provide the cycling duration in the carbon beds that produces effective dampening. Careful design of the two-bed adsorption unit proposed in this study will provide the benefit in both operation and maintenance expense of biofiltration technology for VOC removal from waste gas streams.

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Appendix A. Supplementary data

Supplementary data associated with this article can be found, in the online version, at doi:10.1016/j.cej.2006.10.033.

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