Size, shape and porosity are media physical characteristics, which significantly influence the media's surface reactivity and hydraulic conductivity. Thus, they influence the media’s ability for phosphate sorption. Filter media acts as a vital factor in biofilm growth, phosphate sorption, oxygen substrate mass transfer and is an important influence on the hydraulic characteristics.

3.2 pH

The pH of water determines the solubility (amount that can be dissolved in the water) and biological availability (amount that can be utilised by aquatic life) of chemical constituents such as nutrients. The nitrification process causes reduction in alkalinity value; this will cause decrease in the pH value and finally the nitrification process will be limited or stop completely. The pH value is essential to maintain the nitrification process; the optimum pH for nitrification range from (8 to 9), but as a result of this process will affect the pH value. The pH in the biofilm will be less than its value in the water or wastewater. The sensitivity of nitrifying bacteria and adsorption process to the pH variation shows that the nutrient removal is strongly dependent on the pH of the effluent at the filtration system. Al/Fe metal oxides are positively charged at low pH and negatively charged at high pH values. As phosphate is an anion, adsorption is greatest at low pH and decreases with increasing pH (Klimeski et al., 2012). At high pH, phosphate is adsorbed to surfaces of CaCO3; precipitation of Ca-phosphate is efficient at alkaline pH. Several researchers have studied the influence of pH on phosphate sorption, the findings tabulated in table 1.

Table 1. pH Influence On Phosphate Removal

Media type	pH	Phosphate removal	Reference
Nanoscale zerovalent iron	3 to 12	35 -98 mg p/g	(Wen et al., 2014)
high-surface-area Fe/Mn-hydroxide sorbent	4 to 9	25% to 99%	(ZENG et al., 2008)
Sand	7.70 to 9.57	16.5% to 26.2%	(Del Bubba et al., 2003)
Iron-oxide-coated sand (IOCS-1)	2.4 to 9.5	0.24 to 0.18	(Jianbo et al., 2009)
Iron-oxide-coated sand (IOCS-2)	2.4 to 9.5	0.019 to 0.012	(Wen et al., 2014)

3.3 Operation condition

The variation in the operation process plays a crucial role in the filter performance. Flow properties in filtration systems are affected by the input ratio of air/ liquid, along with the media. in addition, the hydrodynamic characteristics have an effect on the structure of biofilms physically and physiologically (Wilderer et al., 1995), and the phosphate adsorption. In general, shorter hydraulic retention time (HRT) corresponding to high flow rates of water and air will create a strong shearing force. Thus, microorganisms will be affected significantly. This is expecially truefor nitrifying bacteria, which have a gentler reproduction rate and a longer generation period. Probably this may produce a thinner biofilm in the granular media. At longer (HRT), the biofilms are thicker compared with biofilms under shorter (HRT), but the slower wastewater flow rate can lead to lower organic loads, and may negatively affect the bacterial activity. As long as (HRT) is kept under an appropriate controlled value, the bacterial activity can be improved (Han et al., 2009) and the biofilm thickness can stay in a steady state. Table 2 illustrates the influence of HRT on ammonium removal. Contact time between wastewaters and filter media, which acts as phosphate sorption, is highly important; the reaction kinetics of sorbent surface relate to the required contact time that retained phosphate.

3.4 Dissolved Oxygen DO

Several researchers state that the removal efficiency of ammonium is higher at the bottom of the filters because the concentration of the dissolved oxygen (DO) is high and also there is more organic matter available at the bottom. This will provide a better environment for the growing of heterotrophic bacteria; therefore, the biofilm becomes thicker'. However, the DO concentration and the biomass concentration decrease with the media height of the reactors. A specific air–liquid ratio is an essential condition to guarantee normal operation in biological wastewater treatment, as aeration is used during the treatment. An appropriate dissolved oxygen (DO) concentration was suitable for growth of nitrifying bacteria, thus nitrification process was strengthened. The findings in table 2 clarify why the removal of NH3–N increased as air/liquid ratio increased. Suitable air/liquid ratio can effectively remove the aged biomass growing on the surface of filter media, and enhance generation of nitrifying and denitrifying bacteria with high activities of the two processes. However, excessively high air/liquid ratio can create strong air-shearing forces that cut the biofilm and this resulting loss of biomass causes reduction in the thickness of the biofilm, which will lead to a decrease in the removal efficiency of organic substances. In addition, excessively high air/liquid ratio is not cost-effective (Li et al., 2010). The required air amount for treatment is related to the pollution load, the transmission efficiency of oxygen and the rate of the biomass endogenous respiration (Robinson et al., 1994). On the other hand, when the aeration rate is excessively high, scouring of the biofilm and the solids removal efficiency will decline (Pearce, 1996).

3.5 Temperature

Temperature is another factor that has a significant effect on the biological activity of the nitrifying bacteria. The sudden variation in the temperature has a negative effect on the nitrifying bacteria and most other bacteria. The sudden increase in temperature lowers the biological growth rate; on the other hand, the sudden decrease gives a severe drop in the biological activity. If the temperature reaches the range (50-60℃), the nitrifying process does not take place.

Many other environmental factors that have a significant effect on the nutrient removal process should be taken into account to achieve a deep understanding of the overall environmental conditions affecting the removal performance.

Table 2. Ammonium removal efficiency and other parameters for different filter materials

Media type	**Wastewater type**	**DO**	**HRT**	**Ammonium removal efficiency**	**Reference**
Granular activated carbon (GAC)	textile wastewater	2.4 to 6.1mg/L	0.13 to 0.78m/h	68% to 90%	(Liu et al., 2008)
Blast furnace dust clay sodium silicate ceramic particles (BCSCP)	brewery wastewater	3:1- 6:1 A/L ratio	2-8 h	81.9% to 93.2%	(Li et al., 2010)
Water quenched slag particles (WQSP)	municipal wastewater	>4.00 mg/L	1 h to 5 h	81.7% to 95.3%	(Feng et al., 2012)
Haydite	municipal wastewater	> 4.00 mg/L	1 h to 5 h	73.1% to 87.8%	(Feng et al., 2012)
clay ceramic particles (CCP	municipal wastewater	7.5:1 A/L ratio	0.37 h to 1.5 h	39.1% to 86.0%	(Zhao et al., 2009)
Sludge-fly ash ceramic particles (SFCP)	municipal wastewater	7.5:1 A/L ratio	0.37 h to 1.5 h	63.2% to 88.9%	(Zhao et al., 2009)

4. Conclusion

The International and continental tendency to enact legislation that protect the water bodies from eutrophication bring the attention to develop an appropriate treatment process for nutrient removal, as a result of the high proportion of nutrients released from WWTP. Nitrogen in wastewater is originally present in the form of organic nitrogen and ammonia. Orthophosphates are dominating other phosphorus compounds in domestic (municipal) wastewater. The necessity of applying various treatment types like biological, physio-chemical or a combination, makes Dynasand® a promising technology in wastewater treatment. Understanding the contaminants' behaviour and their removal mechanism is important to provide a flexible starting point towards improving the filtration system performance. Biological Nitrogen treatment using filters is receiving more interest worldwide. In addition, the application of phosphorus sorbing materials PSM offer a possible solution for treating municipal wastewater using upflow filters. The chemical composition of the filter media is a key factor of phosphate removal chemically. PSMs can be classified according to their chemical composition; metals, mainly Fe/Al-containing materials or materials containing soluble (Ca and Mg). Physical properties of filter media particles that include size, shape and porosity significantly influence biofilm growth, phosphate sorption, oxygen substrate mass transfer and an important influence on the hydraulic characteristics. Moreover, pH, dissolved oxygen, temperature and operational conditions should be taken into account as they have significant effect on nutrient removal using upflow filters.

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Hydrogen Peroxide-Based Wet Oxidation to Eliminate Contraceptive Hormone Residues from Pharmaceutical Wastewater

Patnaree Sirinukulwatana[1]; Prapaporn Kunpunya[2]; Pattarin Supanivatin[3];
Kenneth W. Foster, Ph.D.[4]; and Aluck Thipayarat, Ph.D.[5]

[1]Dept. of Food Engineering, Faculty of Engineering, King Mongkut's Univ. of Technology Thonburi, 126 Pracha-u-Tid Rd., Bangmod, Tungkru, Bangkok 10140, Thailand. E-mail: vpatnaree.siri@yahoo.com
[2]Dept. of Food Engineering, Faculty of Engineering, King Mongkut's Univ. of Technology Thonburi, 126 Pracha-u-Tid Rd., Bangmod, Tungkru, Bangkok 10140, Thailand. E-mail: prapaporn738@gmail.com
[3]Dept. of Food Engineering, Faculty of Engineering, King Mongkut's Univ. of Technology Thonburi, 126 Pracha-u-Tid Rd., Bangmod, Tungkru, Bangkok 10140, Thailand. E-mail: pattarinohotarn@gmail.com
[4]Dept. of Physics, Syracuse Univ., Syracuse, NY 13244-1130. E-mail: kwfoster@syr.edu
[5]Office of Education, Faculty of Engineering, Burapha Univ., 169 Long-Hard Bangsaen Rd., Saensook, Muang, Chon Buri 20131, Thailand; Dept. of Food Engineering, Faculty of Engineering, King Mongkut's Univ. of Technology Thonburi, 126 Pracha-u-Tid Rd., Bangmod, Tungkru, Bangkok 10140, Thailand. E-mail: aluck@eng.buu.ac.th

Abstract

Wet oxidation using hydrogen peroxide (1, 3, and 5% H_2O_2) at raised temperatures has been used to oxidize pharmaceutical wastewater derived from contraceptive pill production. Model wastewater containing ethinyl estradiol (40 μg/ml), levonorgestrel (25 μg/ml), cyproterone (25 μg/ml), gestodene (10 μg/ml), and desogestrel (50 μg/ml) was formulated based on the average concentration of hormones measured in the first wash of an equipment cleaning process. Higher temperatures for wet oxidation ranging from 100 to 150°C were anticipated to enhance the oxidizing capability of this hydroxyl-radical-mediated advanced oxidation. At mild oxidation treatments (1% H_2O_2 at 100°C), only sensitive species showed minor degradation by HPLC chromatogram. Desogestrel was easily removed even with this mild oxidizing, but the other hormones were more stable. Fortunately, higher temperatures generated much higher hormone degradation rates. For instance, 5% H_2O_2 at 150°C destroyed all the hormones in the model wastewater within 5 min. We assume highly reactive hydroxyl radicals produced the rapid mineralization of the spiked hormones.

INTRODUCTION

Steroidal hormones belong to a group of endocrine disruptors categorized by their origins, either natural (estradiol, estrone, testosterone, estriol) or synthetic hormones (ethinyl estradiol). A weak estrogen (e.g., estriol) excreted in the urine of pregnant women as well as some synthetic estrogens (e.g., mestranol or MeEE2 and ethinyl estradiol or EE2) can occasionally be detected from human sources (Barel-Cohen et al. 2006). Trace of these hormones can be detected in the environment and surface waters worldwide through industrial sewage discharge, animal-farming waste disposal, and effluents of municipal wastewater treatment plants (Desbrow et al. 1998; Kuch and Ballschmiter 2001; Ternes et al. 1999). These synthetic steroidal hormones are widely used to make contraceptives. They are of great concern due to their bio recalcitrance and potential impact of endocrine disruption in aquatic environments (Ying et al. 2002; USEPA 2001; Diamanti-Kandarakis et al. 2009).

Even with extremely low levels of these steroid estrogens (e.g., ng/L), these hormones can alter the normal functioning of endocrine systems adversely affecting reproduction and development in the ecosystem (Jobling et al. 1998; Zheng et al. 2014). The steroidal hormones, particularly 17β-estradiol (E2) and 17α-ethinylestradiol, are among the most potent endocrine disrupting compounds. At the level of 1 ng/l, E2 caused the induction of vitellogenin in male trout (Hansen et al. 1998; Purdom et al. 1994). In surface waters, the concentrations of ethinyl estradiol were generally less than that of natural steroidal estrogens. However, the damage caused by ethinyl estradiol in fish was 10- to 50-fold higher than that of E2 and E1 *in vivo* (Segner et al. 2003; Thorpe et al. 2003). The half-life of ethinyl estradiol was longer and it had a tendency to bioconcentrate in body tissues and bile (Lange et al. 2001; Larsson et al. 1999). Many authors suggest that steroidal hormones in the environment may affect not only animal wildlife and humans but also plant species (Shore et al. 1995; Lim et al. 2000). The widespread occurrence of steroidal hormones in ecosystems is well documented (Nakada et al. 2006; Gomez et al. 2007; Esperanza et al. 2007; Hashimoto et al. 2007; Ifelebuegu and Ezenwa 2011).

The removal of steroidal hormones to below detectability cannot be achieved using conventional wastewater treatments (WWTP). The success rate of ethinyl estradiol removal in conventional sewage treatment plants has varied from case to case being reported from 0% to >98% (Ternes et al. 1999). In batch studies using activated sludge treatment, estrone, ethinyl estradiol, and alkylphenols were not completely eliminated over typical treatment times. Only 85% removal of estradiol, estriol, and ethinyl estradiol were able to be consistently achieved where some species seemed to be more resilient than others (Johnson and Sumpter, 2001). Other studies suggested that it took 6 days for ethinyl estradiol to be degraded completely and transformed to unidentified metabolites (Vader et al. 2000). Ethinyl estradiol removal in soil under aerobic and anaerobic conditions required 44 days but was highly dependent on temperature, moisture, and oxygen (Colucci et al. 2001).

Because most WWTPs were not effective in removing the estrogens and other steroidal hormones from wastewater (Leech et al. 2009; Liu and Mizutani 2009), advanced oxidation processes (AOPs) have emerged as an alternative to remove various types of organics including micro-pollutants and steroidal hormones from wastewater. Fenton's oxidation was proposed to be a cost effective method of oxidation and widely applied for wastewater treatment purposes due to its non-toxicity and absence of mass transfer limitations as a result of its homogenous catalytic nature (Gotvajn and Zagorc-Končan 2005; Ifelebuegu and Ezenwa 2011). The application of UV/H_2O_2 advanced oxidation compared to direct UV alone was able to degrade estrogens more effectively and increased removal efficiency from 20 to above 90% (Rosenfeldt and Linden 2004; Zhang and Li 2014; Nasuhoglu et al. 2012). The hydroxyl radical (·OH) was assumed to play a critical role in the removal of endocrine disrupting compounds during UV/H_2O_2 oxidation. Because of the ability of AOPs to produce highly reactive species such as hydroxyl radicals (·OH), the aim of this paper is to test AOPs that use hydrogen peroxide at elevated temperatures to destroy steroidal hormone activity. Also, this paper examines the optimum oxidative conditions to remove steroidal hormones to a non-detectable level as fast as possible from wastewater samples. The steroidal hormones were assayed using standard HPLC protocols used in the pharmaceutical industry.

MATERIALS AND METHODS

Reagents and Chemicals

Ethinyl estradiol, levonorgestrel, cyproterone, gestodene and desogestrel were received from a local pharmaceutical company at the highest available purity (>98%). The hormones were dissolved in methanol at the two-fold concentration (40, 25, 25, 10 and 50 µg/ml, respectively). These stock solutions were mixed with an equal portion of deionized water containing a two-fold concentration of hydrogen peroxide to achieve the target concentration representing the models of pharmaceutical wastewater after mixing. Hydrogen peroxide (50%), methanol and acetonitrile (HPLC grade) were purchased from Chemipan Corporation Co. Ltd. All other reagents were analytical grade and used as received.

Pharmaceutical wastewater models

Industrial wastewater samples from a local pharmaceutical company were collected from multiple points of the cleaning after contraceptive pill production. The cleaning samples were taken from the first wash and collected in amber bottles used within 2 h following sampling. The concentration of each hormone present in this wastewater was determined using an HPLC protocol to formulate the concentration of the model wastewater.

Hormone degradation experiment

To enable H_2O_2 treatment at temperatures higher than the atmospheric boiling point, a stainless steel pressure vessel was fabricated and equipped with a sanitary clamp for quick discharge of treated wastewater sample (Figure 1). The electric heater provides a manual control of hot oil temperature at approximately 190°C. A pressure gauge was used to monitor the steam pressure buildup inside the vessel. Upon reaching the desirable condition of H_2O_2 treatment, the pressure vessel was taken out of the oil bath to cool down in an ice bucket. The release valve was open to ensure there was no pressurized gas or vapor inside the vessel prior to the release of the sanitary clamp and drawing the sample from the vessel. Several temperature set points (i.e., 100, 125, and 150°C) and incubation times (i.e., 0, 2.5, 5.0, 7.5, 10.0 and 12.5 min) were implemented to evaluate the removal of target steroidal hormones.

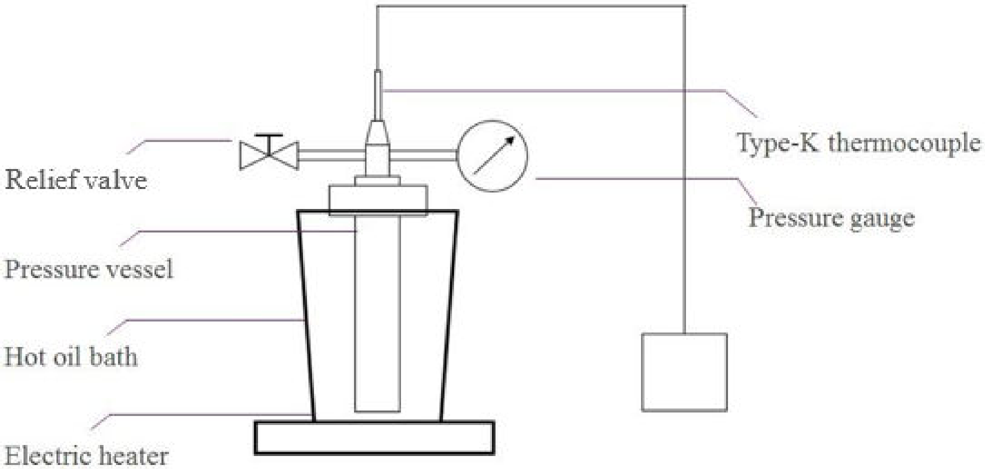

Figure 1. Schematic diagram of the reactor used in UV/H_2O_2 experiments.

Analysis method

To determine the degradation of these five hormones, the first-wash wastewater and wastewater models were analyzed using an Agilent 1200 high-performance liquid chromatograph (HPLC). The wastewater model, which combined ethinyl estradiol, levonorgestrel, and cyproterone, was detected by an HPLC equipped with (C8 reversed phase LC column 4.6 x 150 mm equipped with a 215-nm UV detector). The mobile phase was acetonitrile: methanol: H_2O mixture (35: 15: 45, v/v) at a flow rate of 1.0 mL min^{-1}. The injection volume of the sample was 20 L. The retention time of ethinyl estradiol, levonorgestrel and cyproterone were 5.07, 7.71 and 12.34 min, respectively. The combination wastewater of gestodene and desogestrel utilized a slightly different HPLC setting (Phenyl reversed phase column with a 210-nm UV detector). The mobile phase was acetonitrile: $4KH_2PO$ pH 6.0 mixture (50:50, v/v) at a flow rate of 1.5 mL min^{-1}. The injection volume of the sample was 20 L. The retention time of gestodene and desogestrel was 2.69 and 10.40 min, respectively.

RESULTS AND DISCUSSION

Assessment of sex hormone wastewater

Triplicate samples of first-wash wastewater from multiple types of equipment were evaluated using HPLC. Figure 2 shows an example of a levonorgestrel chromatogram representing the concentration at approximately 3.0 µg/mL of the wastewater generated from the contraception production. The same sampling analysis was performed to determine the averaged concentration for the cleaning wastewater from ethinyl estradiol, levonorgestrel, cyproterone, gestodene and desogestrel production and the averaged first-wash concentrations were approximately 13.3, 3.1, 3.1, 3.3 and 3.0 µg/mL, respectively. To simulate the destruction of these hormones in