



Short Communication

Relative influence of process variables during non-catalytic wet oxidation of municipal sludge



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HIGHLIGHTS

- Fermented municipal sludge was treated by non-catalytic wet oxidation.
- At the beginning, all three process variables had significant effects.
- After 60 min only temperature and oxygen ratio had a significant influence.
- After 60 min interaction of temperature and oxygen ratio had significant effect.

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ABSTRACT

Individual and interactive effects of process variables on the degradation of fermented municipal sludge were examined during wet oxidation. The process was carried out at 220–240 °C using 1:1–2:1 oxygen to biomass ratio and 300–500 rpm stirring speed. Response surface methodology coupled with a faced-centred central composite design was used to evaluate the effect of these variables on total suspended solids, volatile suspended solids and total chemical oxygen demand. Multivariate analysis was conducted for the initial and near completion stages of reaction: 5 and 60 min treatments, respectively. Temperature had the most significant effect on degradation rate throughout. During the initial stage the effect of mixing intensity was less significant than that of oxygen ratio. Mixing intensity did not influence degradation rate at the later stage in the process. During the near completion stage, the interaction of temperature and oxygen ratio had significant effect on sludge degradation.

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1. Introduction

Wet oxidation has been shown to have potential for the treatment of effluents such as municipal sludges which have high concentrations of organic matter or toxic contaminants (Mishra et al., 1995). Wet oxidation involves liquid phase treatment of organic or oxidisable inorganic compounds at elevated temperature and pressure using oxygen (applied as air or pure oxygen gas).

Wet oxidation of individual soluble compounds such as phenol, aromatic alcohols and carboxylic acids in a two-phase reaction environment has been extensively studied by Bhargava et al. (2006), Collado et al. (2010) and Lefèvre et al. (2011). The behaviour of municipal sludge during wet oxidation is less clear due to the complex nature of its components reaction mechanism and the involvement of gas, liquid and solid phases. Wet oxidation of sewage sludge involves two main stages: physical (transfer of oxygen from the gas phase to the liquid phase and the dissolution of hydrolysed solid organic material), and chemical (hydrolysis of

the solid organic materials and reaction between oxygen and organic compounds). These two stages control the overall reaction.

Many variables will affect this process and can act individually and interactively. Whilst oxygen stoichiometry is an important factor, oxygen alone is inadequate, because gas–liquid mass transfer affects the dissolved oxygen. Thus hydrodynamics interact with stoichiometry. Similarly, temperature influences, reaction kinetics, solubility and gas–liquid mass transfer rate (the latter of which is affected by hydrodynamics and the amount of oxygen applied). Clearly these described interactions call for a multivariate approach to unravel the relevant interactions of wet oxidation on organic matter transformations.

Renewed interest in wet oxidation as a treatment application is due to inherent advantages in the handling of municipal sludge and the potential for resource recovery (Debelletfontaine et al., 1996; Foussard et al., 1989; Gielen et al., 2011; Jaroslaw and Roman, 2008; Naoki et al., 2011; Padoley et al., 2012; Ploos van Amstel and Rietema, 1973; Ploos van Amstel, 1971; Strong and Gapes, 2012; Strong et al., 2011; Yang et al., 2010).

Although most of these studies have suggested novel approaches, the assessment and evaluation of the effects of process

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variables has been limited. Consequently, there is a gap in our knowledge regarding the multivariate assessment and evaluation of the wet oxidation of municipal sludge. The present study was designed to contribute more information by evaluating the relative effects of operational variables during non-catalytic wet oxidation of fermented municipal sludge. A clearer understanding of individual and interactive effects of process variables in a wet oxidation system was provided.

2. Methods

2.1. Municipal sludge

Municipal sludge was obtained from the District Council wastewater treatment plant in Rotorua, New Zealand. Of the total sample, 40% was primary sludge and the remaining was secondary sludge. The sludge was fermented at the plant in a 2000 l anaerobic fermenter using a temperature of 35 °C at pH 5.5–6.2. It was then frozen and kept at –20 °C until required.

2.2. Wet oxidation

The temperature of sludge slurry was raised to 4 °C and a sample was homogenised with a magnetic stirrer at the start of each experiment. Wet oxidation was carried out in a Parr Reactor (a 4540 high pressure reactor equipped with a 4848 controller; Parr Instrument Company, US). The reactor (Fig. S1) was equipped with a pre-heated feed tank in which 150 ml of sludge slurry was heated to 90 °C for 5 min with stirring to minimise the temperature gradient associated with transfer to the reactor.

For each experiment the reactor was charged with 250 ml water and pressurised to 20–40 bar with pure oxygen. It was then heated to 220–240 °C before the pre-heated material was introduced by means of pressure difference using pure nitrogen. The initial concentration of solids in the reactor was approximately 1.5 wt%. Using a manual system, 20 ml samples were taken during the initial (5 min) and final (60 min) stages of the wet oxidation process. The sampling tube was flushed with water followed by nitrogen before each sample was collected. Samples were cooled to room temperature immediately to stop further reaction.

A zero time ($t = 0$) sample was prepared outside the reactor with concentration equivalent to that of samples subjected to wet oxidation. The average values of 18 experiments of these untreated samples are: 10300 ± 170 mg/kg total suspended solids (TSS); 8400 ± 140 mg/kg volatile suspended solids (VSS); and 17600 ± 290 mg/l total chemical oxygen demand (tCOD).

2.3. Analysis

TSS and VSS were determined according to standard methods recommended by the American Public Health Association (APHA, 1998).

Total chemical oxygen demand (tCOD) and soluble chemical oxygen demand (sCOD) were determined using an in-house micro-scale method similar to that documented in Standard Methods 5220D (APHA, 1998). Samples were digested with an excess of acidic potassium dichromate solution. The amount of reduced dichromate was determined colorimetrically and was used as an estimate of COD. The tCOD used in this work was calculated based on the following equation:

$$\text{tCOD} = \text{sCOD} + f_{c/v} \cdot \text{VSS}. \quad (1)$$

$$f_{c/v} = \frac{\sum_{i=1}^n \left(\frac{\text{pCOD}}{\text{VSS}} \right)_i}{n} \quad (2)$$

$$\text{pCOD} = \text{tCOD} - \text{sCOD} \quad (3)$$

Where n is the total number of experiments, pCOD is particulate chemical oxygen demands, respectively. The reason is for this approach was that variability in measuring individual tCOD was large when compared with the VSS and sCOD measurements. This caused large variations in the data, preventing a useful statistical analysis of the tCOD parameter. Using a grand mean of the pCOD/VSS yielded a value of 1.46 mgCOD/mgVSS. This is similar to a commonly utilised literature value of 1.42 mg/mg (Tchobanoglous et al., 2003), giving confidence to the approach.

2.4. Experimental design

Response surface methodology (RSM) coupled with a faced-centred central composite design (FC-CCD) was used to investigate effects of the process variables. The Design Expert software, Version 7.1 (Stat-Ease Inc., USA) was used for this purpose.

The independent process variables examined were reaction temperature, oxygen to biomass ratio and mixing intensity. Levels of each variable applied were those indicated published reports of wet oxidation of municipal sludge. The response variables were concentrations of TSS, VSS and tCOD in samples taken 5 or 60 min after commencement of wet oxidation.

A total of 18 experiments provided 8 factorial points, 6 axial points and 4 replications of the centre point. This allowed identification of the broadest possible region in which the standard error of prediction remained relatively consistent. The experimental design matrix established by the FC-CCD is presented in Table S.1.

2.5. Quadratic regression modelling

Experimental data (Table S.1) were used to produce second-order quadratic response surface models for TSS, VSS and tCOD values. Model coefficients and relationships between process variables and response were determined by multiple regression analysis. Separate response surface models were developed for 5 min and 60 min data.

2.6. Analysis of variance

The fitness of the quadratic models obtained from FC-CCD was tested by analysis of variance. Statistical tests (p -value; lack-of-fit; regression coefficient; adequate precision) were used to evaluate:

- Model significance.
- Model fit.
- The effects of individual variables.
- The effects of variable interactions.

The p -value indicates the probability that the variation between conditions may have occurred. A p -value smaller than 0.05 was assumed to indicate a significant model or significant effect. Lack-of-fit indicates the degree of variability of data around a fitted model. If the model does not fit the data, the associated p -value will be greater than 0.10. Regression coefficient (R^2) indicates the extent of variation around the mean values. A model fitting the experimental data exactly would have an R^2 value of 1.0. Adequate precision is a signal-to-noise ratio which compares the range of predicted values at design points with the average prediction error value. An adequate precision value greater than 4.0 is desirable.

Further evaluation of the effects of the process variables on sludge degradation was carried out by means of linear perturbation analysis. This allowed comparison of the effects of all the variables at a particular point in the design space.

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