



Hydrothermal degradation of organic matter in municipal sludge using non-catalytic wet oxidation



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HIGHLIGHTS

- Fermented municipal sludge was treated by non-catalytic wet oxidation.
- A significant degradation of solids occurred within 60 min.
- 98% reduction in the concentration of oxidisable organics was obtained.
- Reaction temperature and oxygen ratio had significant effect.

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ABSTRACT

Of the treatment and conversion technologies used for municipal sludge, wet oxidation has become an important process owing to the characteristics of water as a reaction medium at elevated temperature and pressure. This study aimed to investigate the non-catalytic wet oxidation of municipal sludge and degradation of solids, organic matter and other compounds. A digested sludge consisting of primary and secondary sludges was subjected to 60 min wet oxidation at temperatures ranging between 220 and 240 °C and oxygen to biomass ratios of 1:1–2:1. Wet oxidation was effective at degrading the organic solids, with maximum degradation of 86% for total suspended solids and 96% for volatile suspended solids. The concentration of oxidisable organic matter was reduced by 98% of the untreated sludge. It was found that reaction temperature has the most significant impact on sludge oxidation.

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1. Introduction

Sewage sludge is the semi-solid residue remaining at the end of a municipal wastewater treatment process. Treated liquor is released directly into an aqueous environment while solids are removed for disposal. Owing to increasing public demand for the sustainable management of waste, reduction of the volume of sewage sludge transferred to landfills is a matter of urgency. There are strong regulatory drivers in New Zealand, as there are globally, to increase resource efficiency, increase reuse, recovery and recycling, reduce emissions of greenhouse gases and decrease waste disposal to landfill.

Technological processes exist to divert sludge from landfills, e.g., composting, anaerobic digestion, thermal processing (incineration, gasification, and pyrolysis) and hydrothermal treatment

(thermal hydrolysis). Wet oxidation is another hydrothermal technology with the potential for the treatment of municipal sludge, being particularly applicable for wastes having high concentrations of organic matter or toxic contaminants. Wet oxidation has advantages over alternative treatment technologies as it avoids the need for a dewatering step prior to treatment (such as with incineration, gasification, pyrolysis) and achieves volume reductions as high as those obtained with thermal treatment but at lower temperatures. Non-catalytic wet oxidation has advantages of no need for catalyst removal and to deal the issues associated with catalyst activation and catalyst poisoning by reaction by-products. In wet oxidation, water vapour in the form of steam and CO₂ make up the largest percentage of the off-gas. Unlike traditional combustion or thermal treatments, the off-gas has negligible amounts of CO, NO_x and SO_x. Wet oxidation of municipal sludge has four major potential benefits: (i) enhancement of fermentation, (ii) degradation and removal of organic compounds, (iii) reduction of mass and volume and (iv) recovery of valuable compounds.

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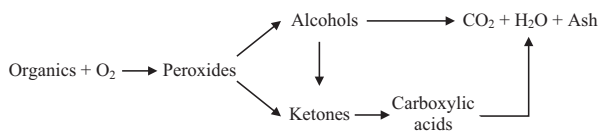


Fig. 1. Schematic diagram showing wet oxidation path of an organic compound [5–7].

Amongst the recoverable compounds are phosphorus, acetic acid and derivable compounds from acetic acid such as calcium magnesium acetate (CMA) [1,2]. It also is able to act as a pretreatment to enhance anaerobic fermentation and digestion to recover methane. Economically, wet oxidation as a treatment is competitive with existing technologies. In a comparison with anaerobic digestion, the economically favourable technology depended upon local conditions, with cost of residue disposal and oxygen costs being principle factors [3] Bertanza et al. (2014) placed a cost of 225–450 €/t_{DS} of treated sludge for wet oxidation and quoted 425 €/t_{DS} by other authors [3].

Wet oxidation involves rapid solubilisation of organic waste and liquid phase oxidation of dissolved organic or oxidisable inorganic compounds at elevated temperature and pressure using oxygen (applied as air or pure oxygen gas). Typical conditions are 150–320 °C under 20–150 bar of pressure for 15–120 min [4].

Previous studies have suggested a simplified pathway for organic matter destruction in wet oxidation based on the formation and destruction of intermediate compounds (Fig. 1) [5–7]. This consists of (i) initial free radical formation; (ii) chain reactions during oxidation of organic compounds; (iii) production of short chain acids, and (iv) ultimate oxidation to CO₂.

Wet oxidation of individual soluble compounds such as phenol, aromatic alcohols and carboxylic acids in a two-phase reacting environment has been extensively studied [8,9].

The behaviour of municipal sludge during wet oxidation is less clear due to the complex nature of its components, the reaction mechanisms involved and the participation of gas, liquid and solid phases. Some research has been undertaken on different aspects of wet oxidation treatment of municipal sludge. As can be seen in Table 1, most of the available literature has dealt with reaction kinetics or has suggested new approaches and focused on the inherent advantages in the handling of municipal sludge and the potential for resource recovery [2,10–26]. There are only a few studies that have investigated transformation of organic compounds into organic acids [2,17,23,24,27–29].

There are also limited investigations focused on the formation and degradation of nutrient compounds (nitrogen and phosphorus) during wet oxidation of municipal sludge [2,15,17,18,21]. If isolatable from the sludge, these nutrients may have valuable utility, for example within the fertiliser industry. These nutrient products offer economic opportunities for recovery and reuse if effectively managed.

In addition to a lack of focus on organic and nutrient compounds, most of these studies have used a slow batch heat-up over time before the desired temperature was reached. This approach could lead to reactions occurring under sub-optimal conditions and the fundamental characteristics of the materials changing prior to the initiation of the wet oxidation conditions under study. Therefore, it is anticipated that by injecting material in a pulse dosing fashion into a system already at temperature, the reactions of interest can be better studied as they occur under the described thermal conditions.

Thus, there is a gap in our knowledge regarding the degradation and removal of organic compounds during wet oxidation of complex mixtures like municipal sludge. The present study was designed to investigate the impact of temperature and availability

of oxygen on the removal of organic materials during non-catalytic wet oxidation of municipal sludge. Our hypothesis is that in non-catalytic wet oxidation, reaction temperature and oxygen both are significant parameters influencing concentrations of suspended solids, organic acids and nitrogen species; and chemical oxygen demand.

In order to avoid the complexity of the potential reactions that may occur during a heat-up process, injection at reaction temperature was used.

2. Materials and methods

2.1. Materials

An anaerobically digested municipal sludge sample was obtained from the Rotorua District Council wastewater treatment plant in Rotorua, New Zealand. This sample consisted of 40% (v/v) primary sludge and the remaining 60% (v/v) was secondary sludge. After sampling, it was frozen and stored at –20 °C until required.

Oxygen (>99.5%) and nitrogen (>99.7%) gas and ultra-zero grade air (Pressure ≤ 200 kPa) were obtained from BOC Limited (New Zealand).

Analytical reagents used in the course of the experiments were potassium dichromate 99.9%, hydrochloric acid 0.8%, sodium carbonate, formic acid 98%, butan-1-ol 99.5%, sodium nitroprusside >99.5% (BDH, Belgium), mercury (II) sulphate, silver sulphate 97% (Hopkin & Williams, UK), sulphuric acid 95–97% (Merck, New Zealand), potassium hydrogen phthalate (Riedel de Haen, Germany), acetic acid >99%, propanoic acid >99%, iso-butyric acid >99% (Merck, New Zealand), N-butyric acid >99%, DCIC sodium dichloroisocyanurate 96% (Aldrich, Germany), sodium hydroxide 97% (Univar, USA), sodium citrate dehydrate (Fisher Scientific, USA), disodium ethylenediamine tetraacetate, sodium dodecyl sulphate (H&W, USA), crystalline phenol >99% (Sigma, USA) and ammonium molybdate tetrahydrate (J.T. Baker, USA).

2.2. Wet oxidation

The temperature of sludge 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. 2) was equipped with a pre-heated feed tank in which 150 mL of sludge slurry was heated to 90 °C and was held constant 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 between 20 and 40 bar with pure oxygen to obtain an oxygen to biomass ratio of 1:1–2:1. These ratios were calculated based on the stoichiometric oxidation potential using an assumed biomass composition of CH_{1.8}O_{0.5}N_{0.2}. The reactor 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 from the reactor. The sampling tube was flushed with water followed by nitrogen before each sample was collected. Samples were quickly cooled to room temperature by passing through a cooling coil to stop further reaction.

Face-centred central composite design (FC-CCD) response surface methodology (RSM) was used to design the experiment. As shown in Table 2, the independent process variables examined were reaction temperature and oxygen to biomass ratio. The response variables measured in the samples taken 60 min after

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