



## Optimization of radiofrequency-oxidation treatment of dairy manure



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### ABSTRACT

A novel technology involving radiofrequency heating and hydrogen peroxide (RF/H<sub>2</sub>O<sub>2</sub>) was used for the treatment of dairy manure. RF/H<sub>2</sub>O<sub>2</sub> process efficacy was affected by power intensity, hydrogen peroxide dosage and holding time. The optimal conditions for nutrient release and solids disintegration were determined: for orthophosphate release, power intensity of 3 kW/L (kW/L substrate treated), holding time of 65 min and H<sub>2</sub>O<sub>2</sub> dose of 1%; and power intensity of 1.9 kW/L, holding time of 53 min and H<sub>2</sub>O<sub>2</sub> dose of 1.2% for soluble chemical oxygen demand concentration. The results indicated that the treatment efficacy of radiofrequency process was comparable to microwave process. However, the radiofrequency process required a longer holding time than microwave process. Nevertheless, energy required by the radiofrequency was lower than microwave as the radiofrequency system was capable of handling higher volumes of manure with higher solids content at a given time.

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### Introduction

Dielectric heat is generated via interaction of dielectric materials with electromagnetic radiation; microwave spectrum and radiofrequency spectrum are generally used for this purpose [1]. The main advantages of dielectric heating are: uniformity of heating and precise control of process temperature. Dielectric heating using microwave frequency was a well established process for environmental applications: it was used for reducing sludge volume, improving dewaterability and bio-digestibility, enhancing nutrient release and pathogen destruction, and stabilizing heavy metal in the wastewater treatment industry; and it was applied in the dairy producing industry for reducing manure solids content, releasing phosphorus and magnesium, and improving effluent quality. A combination of heating and chemical addition for the treatment of wastewater proved to be more effective than heating alone, as indicated in the microwave enhanced advanced oxidation process (MW/H<sub>2</sub>O<sub>2</sub>-AOP) [2–10].

It was thought that a combination of RF heating and hydrogen peroxide (RF/H<sub>2</sub>O<sub>2</sub>) may also be as effective as MW/H<sub>2</sub>O<sub>2</sub>-AOP for the treatment of sewage sludge, manures, and others. RF are at the low-energy end of the electromagnetic spectrum, however, it allows for a greater depth of penetration than microwave due to a longer wavelength; therefore, RF can treat larger volume of materials than microwave process at a given time, as well as materials with high solids content [11]. This study was to explore

the feasibility of the RF/H<sub>2</sub>O<sub>2</sub> process as part of dairy manure management and resource recovery system for the dairy producing industry. Dairy manure contains a rich resource of carbon, nitrogen, phosphorus, and minerals, which can be recovered as useful products [12]. However, most of this resource is not soluble, and a pretreatment step is required prior to any resource conversion process. This study was to identify the significant factors affecting the RF/H<sub>2</sub>O<sub>2</sub> process, and to determine an optimal operating condition for achieving a high degree of solids disintegration and nutrient release.

### Material and methods

#### Apparatus

A 6 kW radiofrequency oven (RF) was used in this study. The RF electrodes were housed in an aluminium box suspended on polypropylene insulators. A transmission line connected the generator output to the oven electrode. The system had a capacity of accommodating up to 9 L of sample in a single run at an operating temperature of up to 95 °C. Commercially available microwave safe plastic containers were used as reaction vessels, with aluminium plates moulded to their bottom surface. The vessels were covered with an aluminium lid that was attached to another aluminium base plate with Teflon spacers in between. The temperature was measured using a Neoptix fibre optic probe inserted into the container through a hole in the lid. The aluminium lid can be removed from the container, without

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removing the Teflon spacers and base plate, when experiments are not performed.

### Substrate

Dairy manure was obtained from the Dairy Education & Research Centre, University of British Columbia in Agassiz, British Columbia, Canada, and stored at 4 °C in the Department of Civil Engineering Labs. Two varieties of dairy manure were used in the first part of this study (part A): the solid portion of dairy manure obtained after solid–liquid separation for tests 1 and 2, and whole manure (un-separated) for test 3. Whole manure was used for the second part of the study (Part B). Dairy manure contained large amounts of sand, bedding material, as well as undigested lingo-cellulosic materials. For the separated dairy manure used in Part A, distilled water was added to the samples and subsequently decanted to remove a large quantity of sand resulting in 4% total solids (TS). Whole manure with TS of 8.1–12.2% was used for part A and B, respectively.

### Experimental design

#### Part A

Three litres of dairy manure was used for the RF process with or without hydrogen peroxide. The runs were carried out in three 5 L vessels. The manure samples were acidified using sulphuric acid to pH 4.0. This was essential as substantial amounts of orthophosphate were released into solution only in acidic condition, preferably below pH 4.0 [13]. After addition of acid, the samples were subjected to RF heating held at 90 °C for 20, 30 or 50 min. Initially, the power control was set to a desired setting level until the manure samples reached 90 °C after which they were held at that temperature for the desired holding time. The experimental design used for the study is given in Table 1.

#### Part B

The surface response design study was carried out in three 1.5 L vessels, and a sample volume of 600 mL was used. Using a statistical program, MINITAB, version 16.0, surface response methodology using the Box–Behnken design was chosen for the experiments [14]. This methodology was chosen to refine models after the important factors have been determined. Box–Behnken design usually have fewer design points than other response

surface designs, it only requires 15 trials for three-level three-factors. The design with three factors by default will have three centre points designated as three zeros; replication is usually done only at the centre point (Table 2). The actual variables in their natural units of measurements were centred and rescaled to the range from +1 to –1. All the patterns in Table 1 included zeroes for one of the factors and a plus or minus combination for the other two factors.

Three factors with three levels per factor, namely, power intensity (2, 2.7 and 3.3 kW/L), holding time (20, 50 and 80 min) and hydrogen peroxide dosage (0.5, 1 and 1.5%) were chosen to study the response (Table 2). The response was expressed as the concentration of soluble parameter, orthophosphate (*ortho*-P), and soluble chemical oxygen demand (SCOD) in the treated solution. An input power level of 50, 60 and 70% was set in the RF unit, which was equivalent to power intensity of 2, 2.7, 3.3 kW/L of manure treated, respectively; a triplicate set for each trial was used. Heating ramp rate was controlled by power intensity. The samples were held at 90 °C for all the experimental trials.

### Chemical and statistical analysis

The initial and treated dairy manure samples were centrifuged at 15,000 rpm for 10 min first, and then their supernatants were extracted. SCOD, orthophosphate, ammonia, volatile fatty acids (VFA) and metals in the soluble portion of the dairy manure were measured. All of the chemical analyses were carried out following the procedures outlined in Standard Methods [15]. For orthophosphate analysis, the initial dairy manure samples were determined at 0.5% TS to ensure correct measurement [16]. Both initial and treated dairy manure were also analysed for TS, total COD (TCOD), total phosphorus (TP) and total Kjeldahl nitrogen (TKN). *Ortho*-P, ammonia, TP and TKN were determined by a flow injection system (Lachat Quik-Chem 8000 Automatic Ion Analyzer, Lachat Instruments, USA). A Hewlett Packard 6890 Series II gas chromatograph, equipped with a flame ionization detector (FID), was used to measure VFA. Volatile separation was accomplished with an HP free fatty acid phase column. Calcium (Ca), magnesium (Mg), sodium (Na) and potassium (K) were determined using a Varian Spectra 220 Fast Sequential Atomic Absorption Spectrometer.

**Table 1**  
Experimental results of Part A study.

Sample	Power intensity (kW/L)	RF holding time (min)	H <sub>2</sub> O <sub>2</sub> dose % (v/v)	Total solids (%)	<i>Ortho</i> -P (mg/L)	Ammonia (mg/L)	Total COD (g/L)	Soluble COD (g/L)	VFA (g/L)
Set 1 (separated manure)									
Raw				4.3 ± 0.2	43 ± 1	653 ± 2	29.9 ± 8.7	6.4 ± 0.4	0.5 ± 0.1
1.1	2	30	0	3.5 ± 0.7	148 ± 3	750 ± 12	23.5 ± 1.2	6.5 ± 0.8	1.6 ± 0.1
1.2	2	30	0.5	2.7 ± 0.2	160 ± 4	816 ± 16	6.2 ± 5.2	6.1 ± 1.0	1.7 ± 0.03
Set 2 (separated manure)									
Raw				4.5 ± 0.4	56 ± 4	634 ± 17	37.2 ± 9.4	3.1 ± 0.3	2.1 ± 0.4
2.1	2	20	0.5	4.2 ± 0.2	127 ± 6	736 ± 6	32.4 ± 2.7	3.9 ± 0.7	2.2 ± 0.0
2.2	2	50	0.5	3.8 ± 0.1	127 ± 5	737 ± 10	24.9 ± 16.3	4.3 ± 0.8	2.3 ± 0.0
2.3	2	20	1	4.3 ± 0.1	116 ± 3	673 ± 7	28.1 ± 14.9	8.4 ± 0.4	2.3 ± 0.0
2.4	2	50	1	4.4 ± 0.2	119 ± 2	737 ± 17	32.5 ± 9.8	6.1 ± 0.1	2.3 ± 0.0
Set 3 (whole manure)									
Raw				9.9 ± 0.1	281 ± 34	1250 ± 184	115.4 ± 16.3	24.9 ± 6.2	–
Acidified				10.7 ± 0.1	360 ± 27	1424 ± 120	101.3 ± 3.8	7.3 ± 2.3	6.1 ± 0.6
3.1	2	20	0.5	8.4 ± 0.0	432 ± 15	1627 ± 58	82.4 ± 0.6	30.7 ± 8.1	6.9 ± 0.3
3.2	2	50	0.5	8.1 ± 0.2	508 ± 8	1885 ± 93	58.1 ± 4.5	20.7 ± 1.9	7.2 ± 0.2
3.3	2	20	1	7.9 ± 0.9	550 ± 10	1900 ± 33	56.2 ± 8.1	22.9 ± 1.1	7.1 ± 0.1
3.4	2	50	1	–	430 ± 18	1707 ± 30	80.9 ± 9.9	25.2 ± 0.4	6.8 ± 0.2

Note: All samples were acidified to pH 4.0 before RF/H<sub>2</sub>O<sub>2</sub> treatment.

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