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A RESPIROMETRIC METHOD TO STUDY BIODEGRADATION KINETICS OF MIXED SLUDGE AND WOODY BULKING AGENT

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ABSTRACT

A respirometric method was set up to study kinetics of biological reactions involved in the composting of solid organic wastes. Two types of sludge were mixed with pine bark. Oxygen consumption rates of these mixtures were monitored during 10 - 20 days, using in a 10 litre respirometric cell kept at constant temperature and moisture. Repeatability of the measurement was demonstrated. The influence on kinetics of the type of sludge and temperature were tested.

INTRODUCTION

The composting process includes two major phases. The first one, called the "active phase", develops degrading reactions: dissolved organic matter is used as carbon and energy source by microorganisms for their metabolism. During the second phase of the composting process, called the "curing phase", organic macromolecules such as humic substances are synthesised. As we already noted microbial reactions form the central phenomena of the "active phase" of the treatment. These reactions involve oxygen consumption, and heat, water and carbon dioxide production. Kinetics of organic matter degradation and quantity of degraded matter are directly linked with biomass growth kinetics (Bailey and Ollis 1986):

$$r_x = \mu X \quad \text{and} \quad r_s = \frac{1}{Y_{x/s}} \cdot \mu X$$

andwith: r_x : biomass growth kinetics, μ : biomass specific growth rate,
 X microorganisms concentration,
 r_s : substrate's degradation kinetics, $Y_{x/s}$: biomass yield

There is also a relationship between biomass growth, substrate degradation and oxygen consumption:

$$r_{O_2} = (1 - Y_{x/s}).r_s = [(1-Y_{x/s})/Y_{x/s}].r_x$$

with r_{O_2} : oxygen consumption kinetics.

Moreover, biological activity is largely influenced by environmental conditions such as oxygen availability, temperature, moisture or pH of the reacting medium. As all the above micro-processes are involved in the treatment and interfere with each other, they are quite difficult to characterise on the sole experimental basis obtained from composting trials. Thus, modelling the biological reactions of the active phase of composting could be very

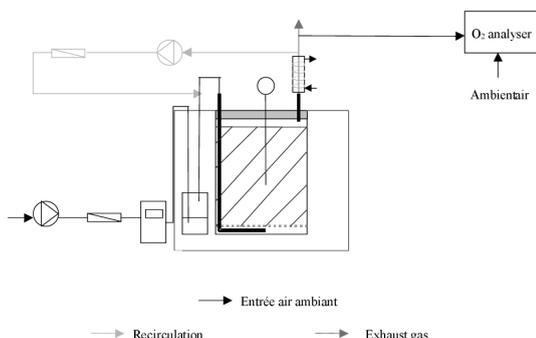
useful to understand and to predict the behaviour of various substrates treated by this process.

Knowledge of the kinetics of biological reactions and of the influencing environmental parameters is then essential to reach this objective. If biomass or substrate concentrations are difficult to measure instantaneously, biological reactions can be studied through respirometric methods by monitoring instantaneous oxygen consumption rate. Indeed, the respirometry is the measurement and interpretation of the biological oxygen consumption under well-defined experimental conditions (Spanjers, Vanrolleghem et al. 1998). Numerous respirometric methods are used to characterise organic matter composition and biodegradation in liquid wastes (wastewater, slurries, etc.). The aim of this study was to develop one of these methods to the special case of a solid mixture of sludge and bulking agent, in which homogeneous oxygen supplying may be a problem. Then, the influence of the type of substrate and temperature was studied.

MATERIALS AND METHODS

Respirometric system design

Figure 1: respirometric apparatus



This system was a closed vessel reactor for the solid organic substrate. The respirometric system could be used as a gas opened system (continuous injection of ambient air and continuous air exit) or as a gas closed system (only recirculation of initial gas volume).

Respirometric measurement's specificity

In order to work without constraint on oxygen availability to microorganisms, oxygen gas concentration needed to be sufficient and uniformly distributed within the reacting cell. Aguilar-Juarez (2000) showed that an oxygen gas concentration greater than 6 % was sufficient to maintain aerobic conditions during the biodegradation of solid organic matter. This condition was respected during our experiments. According to the reactor engineering theory, a perfectly agitated gas flow provides a homogeneous gas concentration in the reacting vessel. Separate experiments were performed to verify this theory.

Temperature and moisture were maintained constant within the cell throughout the measurements.

The respirometric system (figure 1) consisted of:

- a 10 litre glass cell, hermetically closed, filled with the solid organic substrate
- a thermostatic water bath
- a Pt 100 probe measuring temperature in the cell
- a pump to continuously provide ambient air at the bottom of the reacting cell
- a gas volumetric counter
- a pump to recirculate the gas phase
- a gas humidifier and a water condenser
- a paramagnetic oxygen gas analysing device

Substrates

Two types of sludge were considered: (1) a wastewater treatment sludge coming mainly from the agro-food industry effluents (Dry matter (DM) = 17%, Organic matter (OM) = 82,8% DM) and (2) an urban wastewater treatment sludge stabilised before composting through six weeks of anaerobic digestion (DM = 19%, OM = 62,5% DM). In order to work with constant sludge's quality, the sludges were frozen. They were defrosted at 4°C during 48h before each experiment.

Pine bark was used as bulking agent. Mixtures comprised a sludge to bulking agent mass ratio of 1/1 (W/W).

Experimental conditions

Gas phase hydrodynamic was controlled by injecting a pulse of methane, as a tracer gas, in the gas closed respirometric system through the recirculation loop. The cell was filled with sludge (1) and the pine bark mixture. Evolution of methane concentration was monitored at 36°C under four recirculation flow rates: Q, 1.5 Q, 2 Q, 2.5 Q (with Q: minimal gas recirculation flow rate).

Repeatability of measurement was tested by comparing three experiments (A,B,C) conducted on sludge (1) and pine bark mixtures at 36 to 39°C: twice on the same date (A and B) and once one month later (C).

The influence of the sludge type was studied by comparing experiments with sludge (1) and pine bark mixture (DM = 47%) and sludge (2) and pine bark mixture (DM = 51%). These experiments were monitored at 36°C.

The influence of the temperature was tested by comparing oxygen consumption rates in sludge (1) and pine bark mixture (DM = 47%), once at 24°C and once at 36°C.

RESULTS AND DISCUSSION

Method validation

1. Gas phase hydrodynamic

Figure 2 shows methane concentration evolution after a pulse injection in the gas recirculation loop.

Whatever the flow rate, the evolution of the methane concentration in the reacting cell was similar. This evolution was characteristic from tanks in series flow pattern (Levenspiel 1999). That is to say that the gas flow was perfectly homogeneous in the cell after a few minutes. Moreover, final methane concentration enabled the calculation of the gaseous

Figure 2: Mixing flow pattern as a function of recirculation flow rate

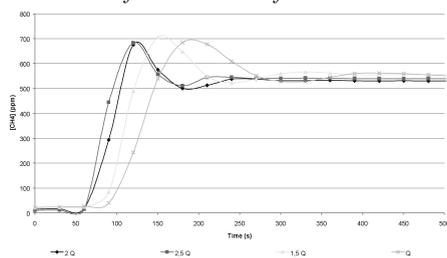
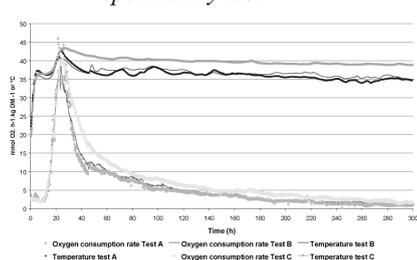


Figure 3: Oxygen consumption kinetics for repeatability test



volume in the respirometric system. This volume was approximately the same as the one calculated on the basis of the characteristics of the apparatus and the porosity of the solid substrates. Thus, no dead volume was detected in the system. Figure 2 also shows that mixing time decreased with increasing flow rate. We chose to work at the fastest recirculation rate that was 7,5 times higher than the ambient air flow rate, imposed when considering the gas opened respirometric system.

2. Repeatability study

The three compared kinetic curves show the same profile (figure 3). After approximately ten hours of latency, an exponential increase of the oxygen consumption rate was observed. This increase lasted ten to fifteen hours and then an abrupt decrease occurred. With time the rate of decrease became slower until constant oxygen consumption rate was reached.

Very good repeatability was observed during the increasing phase of the curve. The variation between the three maximum oxygen consumption rates was around 8 %. In the second part of the curve, the decrease in test C was slower than in test A and B. Global oxygen consumption varied by 18 % between the three experiments. Although the substrates were exactly the same (i.e. the sludge was conserved by freezing), initial heterogeneity of sludge may explain this difference. Consequently, as the behaviour of the curves was similar, good repeatability of the test was admitted.

Respirometric measurement interpretation and study of influencing parameters

The particular shape of the respirometric curves was comparable to the one obtained during respirometric studies of raw wastewater (Kappeler and Gujer 1992; Sperandio 1998). Interpretation of these curves is as follows. After a time lag, biomass consumes oxygen to degrade the most easily biodegradable substrate and to sustain its own growth requirements. With the increasing biomass, the oxygen consumption accelerates until nearly all the easily biodegradable substrate is degraded. A break in the exponential growth is then observed. Biomass continues to develop by hydrolysing and then degrading slowly biodegradable substrate(s). As a consequence, oxygen consumption rate decreases regularly up to complete disappearance of the biodegradable substrates. This explanation has been verified when no other limitation than substrate availability occurs (oxygen is always sufficient) and has been used to explain our results.

1. The influence of sludge type

Respirometric behaviour of two types of mixture is presented in figure 4. No latency was observed with sludge (2) and the growth of the oxygen consumption rate stopped after only 3 hours. Total oxygen consumption up to constant rate was approximately half in mixture with sludge (2) to that in the mixture with sludge (1). This result was in agreement with sludges' origins. Indeed the biological pre-treatment of sludge (2) had already consumed a large part of the easily biodegradable organic matter. As the total consumed oxygen is proportional to the quantity of total biodegradable organic matter, it can be assumed that the mixture with sludge (1) contained two times more biodegradable organic matter per kilogram of dry matter than mixture with sludge (2). Thus, with equivalent experimental conditions, respirometry enables the comparison of the substrates' biodegradability. This method may also be used to characterise organic matter quality and treatment efficiency afterwards.

Figure 4: Oxygen consumption rate in function of sludge type

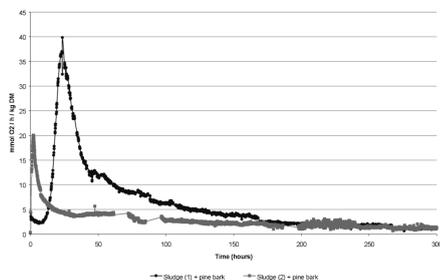
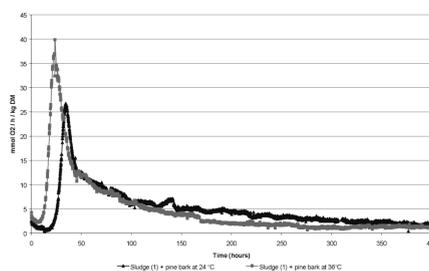


Figure 5: Oxygen consumption rate in function of temperature



2. Influence of the temperature

Oxygen consumption curve profiles (figure 5) were similar for both temperatures. Moreover total quantities of consumed oxygen up to a constant oxygen consumption rate were equal, i.e. the quantity of biodegradable organic matter in both experiments was equal.

Nevertheless, kinetic rates were less rapid at 24°C than at 36°C. When considering exponential growth and decrease, oxygen consumption kinetic curves can be linearised as $\ln r_{O_2} = a.t + b$, where "a" represents a kinetic constant of the curve's growth or decrease. Results of linearised curves' slopes are presented in table 1.

Table 1: Slopes values of linearised respirometric curves at 24°C and 36°C

Experimental temperature (°C)	Maximum consumption rate (mmol O ₂ / kg DM / h)	Growth slope (h ⁻¹)	Decreasing slope (h ⁻¹)
24	27	0,20	-0,0051
36	40	0,23	-0,0103

The exponential growth and decrease of the curves are representative of first order kinetics for oxygen consumption. Our results show that these kinetics were clearly sped up with increasing temperature. Indeed, in this range of values, increasing temperature generally speeds up microbial growth (Bailey and Ollis 1986). As a consequence oxygen consumption rate increases. Moreover with increasing growth, biomass may produce more enzymes to hydrolyse slowly biodegradable organic matter. Then total biodegradable organic matter is more rapidly consumed. Therefore, the oxygen consumption rate, depending on substrate quantity, decreases faster. Such behaviour was observed during our experiments.

CONCLUSION

A respirometric method with a closed vessel and a gas-opened system was validated to study oxygen consumption kinetics occurring in waste mixtures. Constant experimental conditions were verified: constant temperature and moisture, and no constraint on oxygen quantity and flow. The repeatability of the measurements was proven.

This method enabled us to compare substrates' biodegradability and to study the influence of an environmental parameter such as temperature on the kinetics of the reactions.

These results show that this method may be used to study kinetics of biological reactions occurring during the "active phase" of composting treatment. Further experiments have to

be conducted in order to: (a) establish kinetic profiles of different types of waste mixtures; (b) express the influence of environmental parameters; and (c) model kinetic curves as Monod's kinetics and find the associated parameters.

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BIBLIOGRAPHY

- Aguilar-Juarez, O. (2000). "Analyse et modélisation des réactions biologiques aérobies au cours de la phase d'exploitation d'un casier d'un centre d'enfouissement technique." Thèse, Institut National des Sciences Appliquées, Toulouse
- Bailey, J. and Ollis, D. (1986). *Biochemical engineering fundamentals*. Singapore, McGraw - Hill Book Co.
- Kappeler, J. and Gujer, W. (1992). "Estimation of kinetic parameters of heterotrophic biomass under aerobic conditions and characterization of wastewater for activated sludge modelling." *Water Science and Technology* **25**(6): 125-139.
- Levenspiel, O. (1999). *Chemical reaction engineering*. New York, John Wiley and Sons.
- Spanjers, H., Vanrolleghem, P., et al. (1998). *Respirometry in control of the activated sludge process: principles*. London, International Association on Water Quality.
- Sperandio, M. (1998). "Développement d'une procédure de compartimentation d'une eau résiduaire urbaine et application à la modélisation dynamique de procédés à boues activées." Thèse, Institut National des Sciences Appliquées, Toulouse