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## Impact of thermal treatment on the rheological properties and composition of waste activated sludge: COD solubilisation as a footprint of rheological changes



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### HIGHLIGHTS highlights are the second control of the secon

- COD solubilisation follows the same trend regardless of concentration and temperature.
- There are similar decreasing trends of yield stress and infinite viscosity.
- The increase of sCOD is proportional to decrease of yield stress and infinite viscosity.
- The change of rheology and composition of WAS & digested sludge follows the same pattern.

### article info

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The rheological properties of sludge are key parameters in the design and optimisation of wastewater treatment processes. However, these properties are irreversibly altered when the sludge is exposed to heat (e.g. either passing through heat exchangers of anaerobic digesters or during thermal pretreatment process before digesters). Literature showed that thermal pre-treatment of waste activated sludge (WAS) solubilises particulate organic matter in sludge and increases the biodegradability of WAS in anaerobic digesters.

This paper reports the similarity between the kinetic of rheological enhancements due to thermal treatment and the rate of organic matter solubilisation in the WAS when it is subjected to cyclic thermal treatment between 20 and 80  $\degree$ C up to 1 h. This work observed the evolution of rheological characteristics and soluble oxygen demand (sCOD) of 3.5–9.9 wt.% WAS at different temperatures and for different thermal histories (1 h thermal treatment at 50, 60, 70 and 80 $\degree$ C).

Results showed that, the rate of yield stress and infinite viscosity reductions due to thermal treatment (for 1 h treatment at 50, 60, 70 and 80 $^{\circ}$ C) were linearly proportional to the rate of solubilisation of organic matter. Because similar results were observed in the literature with digested sludge, these results confirmed that rheological measurement (e.g. infinite viscosity and yield stress) can be used to predict the level of solubilisation of organic matter in any type of sludge when it is exposed to thermal treatment. Vice versa, the rate of COD changes can be used as an indicator of the rate of rheological changes.

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### Nomenclature



### 1. Introduction

Nowadays, many developed and developing countries use anaerobic digestion process to biodegrade waste such as municipal sewage sludge [\[1\]](#page--1-0). The anaerobic digesters operate either at 35– 40 °C or at 50–60 °C with quite long retention time of sludge (15–45 days). As a result of that the sludge is usually recirculated through the heat exchanger continuously to keep the temperature constant [\[2,3\].](#page--1-0) In addition, Ruiz-Hernando et al. [\[4\]](#page--1-0) has highlighted that the implementation of low-temperature thermal pretreatment of waste activated sludge (WAS) before digesters has a potential to increase the efficiency of the anaerobic digestion process. A thermal pre-treatment of WAS can increase the digestibility of WAS in digester as well as increasing the bio-gas production [\[5\].](#page--1-0)

WAS exhibits a non-Newtonian, temperature dependant rheological behaviour  $[6-8]$ . In addition, WAS is a suspension containing microorganisms which are continuously digesting organic matter and causing rheological ageing effects [\[9\].](#page--1-0) Temperature is known to highly affect the die or disrupt kinetics of biomasses [\[10\]](#page--1-0). Thus, a better understanding of the impact of temperature on the rheology of WAS is required for the design and optimisation of anaerobic digesters, heat exchangers and recirculation loops as well as design of a thermal pre-treatment process before the digesters [\[7,11\]](#page--1-0). In addition to the application of rheology in the mechanical design of equipment used in wastewater treatment processes, the rheology may also be used for the control of actual digestion process [\[12\]](#page--1-0). So, a deep understanding of the impact of temperature on rheology of such a complex system (i.e. non Newtonian, time and temperature dependant suspension) is necessary.

Various modified Arrhenius type equations have been proposed in literature to predict the effect of temperature on rheological properties of sludge  $[13,14]$ . This is however, objectionable since it is well known that thermal treatment of sludge solubilised the organic compounds of sludge [\[15\]](#page--1-0) while Arrhenius type equations are based on the concept of activation energy which assumes material is not changing. So the assumption of handling the same material composition after thermal treatment is not valid for sludge.

It is currently known that low-temperature thermal treatment irreversibly changes the rheology of digested sludge as well as its composition (e.g. solubilised particulate organic matter) [\[16\].](#page--1-0) Furthermore, a linear relationship was found between the kinetics of compositional changes and rheological changes for the duration of thermal treatment of digested sludge [\[12\]](#page--1-0). However there are differences between WAS and digested sludge composition and rheology:

- The COD fractions of WAS and digested sludge are different with regards to its potential of biodegradability and its physical state [\[17\].](#page--1-0) Total COD can be divided into different fractions such as: soluble (readily) biodegradable, soluble non-biodegradable, slowly biodegradable particulate and non-biodegradable particulate matters.
- In terms of rheology, WAS behaves like a colloidal gel material [\[18\]](#page--1-0) while digested sludge behaves like an emulsion [\[19\]](#page--1-0).

In this study, the effect of temperature and thermal history on rheological properties of WAS was investigated. The rate of organic matter solubilisation was measured using COD analysis on both soluble and total phases with the aim of finding a relationship between rheological changes and compositional changes of WAS due to thermal treatment. Then, the response of WAS to thermal treatment was compared to digested sludge.

### 2. Materials and methods

The waste activated sludge was sampled at Mount Martha waste water treatment plant (Victoria, Australia) with original solid concentration of 3.5 wt.%. A vacuum filtration process was used to thicken the sludge to higher solid concentrations (4.7, 6.1, 7.0, 8.3 and 9.9 wt.%). Total solid concentration was measured according to APHA [\[20\]](#page--1-0) standards for wastewater examination.

A stress-controlled rheometer (HR3) from TA Instruments was used to measure yield stress and the flow curve of sludge at different concentrations and temperatures from 20 to 80 $\degree$ C. The rheometer was connected to a water bath capable of heating up the sludge to the test temperature and cooling it down to the initial temperature. Cup and bob geometry was used for the flow curve measurements and a rough cup and vane geometry was used for the yield stress determination. The procedure which was used before each test to erase the shear history of the samples as follows: mixing at low speed until the sample reaches to the test temperature, 5 min pre-shearing at the highest shear rate of actual test, i.e.  $1000 s^{-1}$  and then applying 1 min equilibrium at zero shear rate. The yield stress point was determined using tangent crossover methods from shear stress–deformation diagram [\[21\].](#page--1-0) To do so, the yield stresses were determined at the limit of linear elastic deformation using two fitting lines in log–log diagram of shear stress–deformation. The first line was fitted to the beginning of the curve at low deformation values (linear elastic region) and the second fitting line was fitted to the curve where an abrupt increase in deformation values occurred (flow region). The shear stress value at crossover point was taken as the yield stress point

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