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# HYDROLYSIS RATES OF DOMESTIC WASTEWATER SLUDGE USING BIOCHEMICAL METHANE POTENTIAL TESTS

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## ABSTRACT
***Domestic wastewater treatment can be improved by reducing energy consumption and increasing carbon recovery, which can be achieved using anaerobic digestion of sludge with methane recovery at ambient temperature. Hydrolysis can be a limiting step in anaerobic digestion, and characterisation of hydrolysis rates and process models should improve design and operation of treatment systems. The hydrolysis of primary sludge and secondary sludge were examined using biochemical methane potential (BMP) tests, with the monitoring of volatile solids concentrations and pH values at 25°C and 37°C, and data analysis using MATLAB non-linear least squares curve fitting to a first order hydrolysis model. Low reduction of solids was observed at 25°C compared to 37°C, and higher hydrolysis rates at 25°C than at 37°C. A correlation was observed between the first order model, digestion time and the reduction of solids based on coefficients of determination ($R^2$). Model predictions were close to observed values, and therefore, the model should be reliable in predicting hydrolysis of sludge at 25°C and 37°C.***

***Keywords****: hydrolysis; domestic wastewater; sludge; biochemical methane.*

## 1. INTRODUCTION
Sludge is the material collected through the sedimentation of particulate compounds during wastewater treatment, and its disposal is a critical aspect of domestic wastewater treatment [1]. Conventional wastewater treatment plants, for example activated sludge plants, usually produce two types of waste sludge, which are the primary sludge (PS) and the secondary sludge (SS) [2]. Primary sludge is collected in primary settling tanks in the treatment process, while secondary sludge is the particulate waste after aeration tanks or trickling filters, and is also referred to as waste activated sludge (WAS) for sludge from activated sludge plants. Several researchers have reported less than 50% of organic carbon recovery rates by systems treating domestic wastewater [3]. Anaerobic digestion of sludge from wastewater treatment plants can become a reliable method for carbon recovery in the form of methane [4], and there is a need to advance the understanding of the process in order to ensure efficiency.

Most anaerobic digesters are operated at a fixed mesophilic temperature in order to ensure process stability and efficiency, however recent concerns relating to energy efficiency and climate change has encouraged the consideration for digesters with low energy requirement or without temperature control [5]. A change in process temperature usually causes a change in the physical and chemical properties of wastewater [6 - 7], for example viscosity of liquids is influenced by temperature with high viscosity at low temperatures and therefore, different energy requirements for mixing will exist depending on process temperature [8]. Anaerobic digestion is a complex process which normally involves the following stages: hydrolysis (liquefaction), acidogenesis (acid formation), acetogenesis (acetate formation) and methanogenesis (methane formation) [9].

Hydrolysis is the conversion of the complex biodegradable organic matter into more readily soluble biodegradable matter which can then serve as necessary carbon source for the completion of the anaerobic process [10]. Hydrolysis is considered as a limiting step in anaerobic digestion, due to its slow rate and variations in characteristics of substrates, temperature and pH [10 – 11]. The factors known to influence hydrolysis and anaerobic digestion include substrate characteristics, reactor configuration, operational parameters (for example hydraulic retention), the type of microorganisms present in the

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biomass, and environmental factors, such as temperature and pH value [10].

Generally, secondary sludge has reported hydrolysis rates half the rates reported for primary sludge and the performance of the anaerobic process will be influenced accordingly based on the nature of the substrate used as feedstock [12 - 13]. However, there is a wide range in the values of hydrolysis rates reported in literature, mainly due to different experimental conditions and biomass-to-substrate ratios [14]. The advancement of characterisation of hydrolysis rates and adoption process models based on the kinetics of anaerobic digestion can provide an understanding of hydrolysis behaviour and ensure accurate design and operation of anaerobic treatment of wastewater [15].

Most of the hydrolysis process models proposed in literature are considered to have a major limitation, which is they are usually based on specific experimental conditions, for example very high or very low substrates to microorganism ratio [16]. In a comparison of hydrolysis kinetic models, Vavilin *et al.* [17] concluded that their experimental data fits all the tested hydrolysis models comparatively well and therefore the application of first-order kinetics, which is the simplest way to describe the hydrolysis rate, is acceptable. Vavilin *et al.* [17] recommended the application of a first-order kinetic model to describe hydrolysis rates, where hydrolysis in a batch process can be represented in the form of Equation 1 [17].

$$P = P_o(1 - f_h) + f_h P_o e^{-k_h t} \qquad (1)$$

In (1), P is the concentration of total substrate (mg/L), $P_o$ is the initial concentration of total substrate (mg/L), $f_h$ is the biodegradable fraction of substrate, $k_h$ is the hydrolysis rate constant ($day^{-1}$), t is the time (day) and e is the 2.7182

The aim of this paper is to evaluate the efficiency of the anaerobic hydrolysis of domestic wastewater primary sludge and secondary sludgeatambient temperature. Specific objectives of this paper are the evaluation of the influence of temperature on the efficiency of the hydrolysis process and the relationship of Equation 1 to the hydrolysis process. Consequently, the anaerobic digestion of domestic wastewater primary sludge and secondary sludge were monitored at 25°C and compared with digestion at 37°C using biochemical methane potential (BMP) batch tests.

## 2. MATERIALS AND METHOD

### 2.1 Materials

The substrates and anaerobic biomass used were:

a) A Five litre composite sample of domestic wastewater primary sludge;
b) A Five litre composite sample of domestic wastewater secondary sludge;
c) A Five litre sample of anaerobic digested sludge;

The chemicals and reagents used were:

a) Ammonium Bicarbonate ($NH_4HCO_3$), Sigma Aldrich, UK;
b) Potassium Dihydrogen Phosphate ($KH_2PO_4$), Sigma Aldrich, UK;
c) Magnesium Sulphate ($MgSO_4$), Sigma Aldrich, UK;
d) Iron (III) Chloride ($FeCl_3$), Sigma Aldrich, UK;
e) Calcium Chloride ($CaCl_2$), Sigma Aldrich, UK;
f) Potassium Chloride (KCl), Sigma Aldrich, UK;
g) Cobalt (II) Chloride ($CoCl_2$), Sigma Aldrich, UK;
h) Nickel Chloride ($NiCl_2$), Sigma Aldrich, UK;
i) Sodium Bicarbonate ($NaHCO_3$), Sigma Aldrich, UK;
j) 10M Sodium Hydroxide (NaOH) solution, Sigma Aldrich, UK;
k) Nitrogen gas (99%), Sigma Aldrich, UK;

The major instruments used were:

a) SenSION3 pH probe and meter (Hach Company, Loveland Colorado U.S.A)
b) DR 5000 Hach Lange spectrophotometer (Hach Lange, Salford Manchester, UK)
c) MATLAB curve fitting toolkit (MATLAB R2013a student version, MathWorks, Cambridge, UK)
d) 500 mL glass bottles (Fisher Scientific, UK)
e) 25°C cabinet incubator
f) 37°C cabinet incubator
g) 105°C oven
h) 550°C furnace

### 2.2 Preparation of the BMP Batch Tests

The five litre composite samples of the primary sludge and secondary sludge were obtained from various domestic wastewater treatment plants in Scotland, through the agency responsible for sewerage services in Scotland, Scottish Water. Digested sludge was sourced from the anaerobic digester of Hatton wastewater treatment plant in Arbroath, Scotland, and used as a source for anaerobic biomass. A nutrient solution was also prepared, as recommended by Angelidaki and Sanders [18], containing only micronutrients and trace metals necessary for growth of microorganisms dissolved in distilled water without any substantial amount of organic carbon [18]. The composition of the nutrient medium in this study was: 75 mg/L Ammonium Bicarbonate ($NH_4HCO_3$), 400 mg/L Potassium Dihydrogen Phosphate ($KH_2PO_4$), 5.0 mg/L Magnesium Sulphate ($MgSO_4$), 5.0 mg/L Iron (III) Chloride ($FeCl_3$), 5.0 mg/L Calcium Chloride ($CaCl_2$),

5.0 mg/L Potassium Chloride (KCl), 1.0 mg/L Cobalt (II) Chloride ($CoCl_2$), 1.0 mg/L Nickel Chloride ($NiCl_2$) and 500 mg/L Sodium Bicarbonate ($NaHCO_3$).

The substrates and anaerobic biomass were characterized to determine their initial total solids (TS), volatile solids (VS), pH and volatile fatty acids (VFA) concentrations prior to initiation of the BMP tests. The anaerobic biomass was degassed for 48 hours by incubating at 37°C before the preparation of the BMP tests. 500 mL glass bottles sealed with thick rubber septum and aluminium caps were used for the tests, based on the recommended methodology by Angelidaki *et al.* [19], according to the compositions provided in Table 1, where each mixture was prepared in duplicate bottles and the experiment was carried out for 40 days.

The pH values of the final mixtures were adjusted by carefully adding a few drops of a 10M Sodium Hydroxide (NaOH) solution to each mixture until the pH reading was between 7.51 and 7.88. Then 350 mL of the mixtures were measured into labelled bottles, allowing for a headspace of 150 mL in order to avoid pressure build-up in the bottles once methane production started. The bottles were capped and the headspace was flushed with Nitrogen gas for 2 min to remove oxygen from the headspace, and then placed in 25°C and 37°C cabinet incubators.

### 2.3 Collection of Samples from the BMP Tests

Samples were collected from the BMP tests through the septum cap using Plastipak® 2 mL disposable plastic hypodermic syringes and 21-guage needles (Fisher Scientific, UK). For parameter analysis, samples were collected from each test condition in five 2 mL volumes and mixed to make 10 mL composite samples, in order not to deplete the volumes inside the test bottles before the experimental period elapsed.

### 2.4 Analytical Method

Total solids concentrations were determined based on recommended standard method [20], by drying the samples in an oven at 105°C over 24 hours, while the volatile solids concentrations were determined by igniting the dried samples in a furnace at 550°C for two hours. The measurements were performed in duplicate for each sample, and the average TS and VS was adopted. The pH of the samples was determined using a SenSION3 pH probe and meter (Hach Company, Loveland Colorado U.S.A). VFA concentrations, expressed as acetic acid (mg/L HOAC) within the range of 27 – 2800 mg/L, were determined by spectrophotometry with the ferric hydroxamate method for determination of carboxylic esters [21 - 22], also known as the Montgomery method, using a DR 5000 Hach Lange spectrophotometer (Hach Lange, Salford Manchester, UK). The analysis, defined as Method 8196 in the DR 5000 user manual [23], was performed in triplicates for each sample, and the average of the three measurements was adopted as the VFA concentration for the sample.

### 2.5 Data Analysis

Regression analysis of recorded volatile solids concentrations during the BMP tests was conducted using non-linear least squares fit method to Equation 1 with the MATLAB curve fitting toolkit (MATLAB R2013a student version, Math Works, Cambridge, UK) [24]. Statistical analysis of the data fit to Equation 1 was carried out by the MATLAB curve fitting toolkit using the coefficient of determination ($R^2$), the sum of squares due to errors (SSE) and root mean squared error (RMSE). $R^2$ indicates the square of the correlation between the predicted model values to the initial observed values [25], while RMSE is the root-mean-square error, which is a measure of the differences between values predicted by the model and the values observed [26]. SSE is the sum of squares due to error, which measures the total deviation of the predicted values to the observed values. Data outliers, values outside 95% confidence levels, were removed during the curve fitting process in order to get a fit between Equation 1 and the observed data.

*Table 1: 350 mL BMP tests for domestic wastewater sludge*

| ID | Temp. (°C) | Substrate volume (mL) | Anaerobic biomass volume (mL) | Nutrient solution volume (mL) |
|---|---|---|---|---|
| PS 37°C | 37 | 150 Primary sludge | 100 Anaerobic biomass | 100 |
| PS 25°C | 25 | 150 Primary sludge | 100 Anaerobic biomass | 100 |
| SS 37°C | 37 | 150 Secondary sludge | 100 Anaerobic biomass | 100 |
| SS 25°C | 25 | 150 Secondary sludge | 100 Anaerobic biomass | 100 |
| Blank | 37 | - | 100 Anaerobic biomass | 250 |
| Blank | 25 | - | 100 Anaerobic biomass | 250 |

## 3. RESULTS AND DISCUSSION

Initial characteristics of the sludge samples and anaerobic biomass in terms of total solids (mg/L), volatile solids (mg/L), volatile fatty acids (mg/L acetic acid) and pH values are provided in Table 2.

*Table 2: Characteristics of solid substrates*

| Parameter | Primary sludge | Secondary sludge | Anaerobic biomass |
|---|---|---|---|
| Total solids (g/L) | 28.96 | 32.11 | 18.38 |
| Volatile solids (g/L) | 19.43 | 21.05 | 9.06 |
| Volatile fatty acids (mg/L) | 359.30 | 240.10 | ≈ 0.00 |
| pH | 5.98 | 6.22 | 7.70 |

Note that "≈" in Table 2 indicates values observed were within margin of error of the analysis, and therefore considered as "0.00", which is expected for the anaerobic biomass after degassing for 48 hours. The concentrations of solids in the primary sludge and secondary sludge indicates the sedimentation and dewatering processes in the wastewater treatment systems, instead of the quality of the initial wastewater before the treatment processes and collection of the sludge. After preparation of the BMP tests, the ratios of the mass of volatile solids in the substrates to the mass of volatile solids in the anaerobic biomass in the tests at initiation were 3.2:1 for the primary sludge tests and 3.5:1 for the secondary sludge tests. Figure 1 presents remaining fractions of volatile solids of the substrates against experimental time (days), and the results indicate that the reduction of the solid substrates for all the experimental conditions tested exhibited a trend similar to the model prediction, represented by the 'lines of best fit' in Figure 1.

Most of the reduction in solids was observed within the first ten days of the experiment, during which more than 30% of the primary sludge were reduced (Figure 1). The reduction of the solids for the secondary sludge was lower, about 20%, than the reduction in the primary sludge. However, by the end of the experiment the reduction of primary sludge at 25°C was similar to the reduction of secondary sludge at 37°C, about 40%. The fractions of the substrates retained at the end of the experiment are indications of the inaccessible and non-biodegradable fractions of the substrates. The secondary sludge is expected to have smaller particle sizes than the primary sludge [27], therefore, the secondary sludge should be more accessible to the microorganisms during hydrolysis than the primary sludge. However, with the lower reduction of the solids in the secondary sludge compared to the primary sludge, there is a possibility that other factors, for example biomass to substrate ratio, influenced the hydrolysis of the sludge. From Equation 1, the remaining biodegradable fraction of the substrate depends on the hydrolysis rate and the digestion time, as defined in Equation 2.

$$P_{biodegradable} = f_h P_o e^{-k_h t} \quad (2)$$

In (2), $P_{biodegradable}$ is the biodegradable concentration of total substrate (mg/L), $f_h$ is the biodegradable fraction of substrate, $k_h$ is the hydrolysis rate constant ($day^{-1}$), t is the time (day), and e is the 2.7182.

The exponential component of the equation will increase with a decrease in the value of $k_h t$, therefore large $k_h$ values will yield low retention of biodegradable fractions compared to small $k_h$ values which should yield high retention of biodegradable fractions. A few data points presented in Figure 1 are higher than 1.0, and these values indicate experimental errors, potentially as a result of the sampling method adopted, where needles and syringes were used. Literature reviewed [19, 28 - 31] did not provide specific details of methods for collection of solid samples from closed batch test experiments such as the BMP tests. Researchers have proposed potential modifications to the methodology in order to avoid data errors [22], for example by monitoring the total mass of the system over time. Figure 2 presents the pH values in the tests for the corresponding experimental days, where all the pH values were within a range, 6.50 to 8.00, suitable for anaerobic digestion without any substantial variation from this range, from the second day of the experiment.

During the experiment, the initial period was the hydrolysis and acid forming stage of the anaerobic digestion, and this is represented by the decrease in pH values in the first five days (Figure 2). The pH values did not fall below 5.0, which will have been an indication of process instability and accumulation of acids in the test [32], and the pH values stabilized without any pH adjustment during the experiment. Table 3 presents the summary of the reduction in terms of the fractions of the substrates removed by mass during the BMP test and the predicted reduction by mass using Equation 1, presented as the 'modelled' column.

In Table 3, "Temp" represent test temperatures, "$R^2$" represent the coefficients of determination, "SSE" represent the sums of squares due to errors and "RMSE" represent root mean squared errors of the BMP tests. The observed reduction of volatile solids of the

substrates, Table 3, showed low reduction at 25°Ccompared to 37°C, where for the primary sludge (PS) test, over 55% of the volatile solids were reduced at 37°C, while only 40% reduction was observed at 25°C (Table 3). For the secondary sludge (SS) test, over 33% of the volatile solids were reduced at 37°C, while only 22% reduction was observed at 25°C. From Table 3, the high $k_h$ values for the sludge correspond to the 25°C tests, while the low $k_h$ values correspond to the 37°C tests, however the retained biodegradable fractions were lower at 37°C than at 25°C. This indicates that the rate of hydrolysis at 25°C was faster than at 37°C, even though the degree of hydrolysis was substantially less at 25°C than at 37°C. Furthermore, the degree of hydrolysis of the secondary sludge was substantially lower than the degree of hydrolysis of the primary sludge.

The correlation between the observed solids reduction and the prediction of Equation 1 can be evaluated based on the coefficient of determination ($R^2$) values obtained, which are indications of the usefulness of the model in predicting the process as a function of time [25]. This means if $R^2$ is a value close to 1.0, then Equation 1 is useful in predicting the hydrolysis of the substrate and the length of experimental time is important in determining the reduction in the substrate. While Equation 1 is not useful in predicting the hydrolysis of the substrate and the length of time is not important in determining the reduction in the substrate, if the $R^2$ value is close to 0.0.

Based on the $R^2$ values obtained in Table 3, Equation 1 is appropriate for use in the prediction of the reduction of primary and secondary sludge in batch systems with temperature and biomass conditions similar to the BMP tests in this study. From Table 3, the number of data points is an indication of the number of outlying data points (due to errors in measurements) that were not considered for the regression analysis.

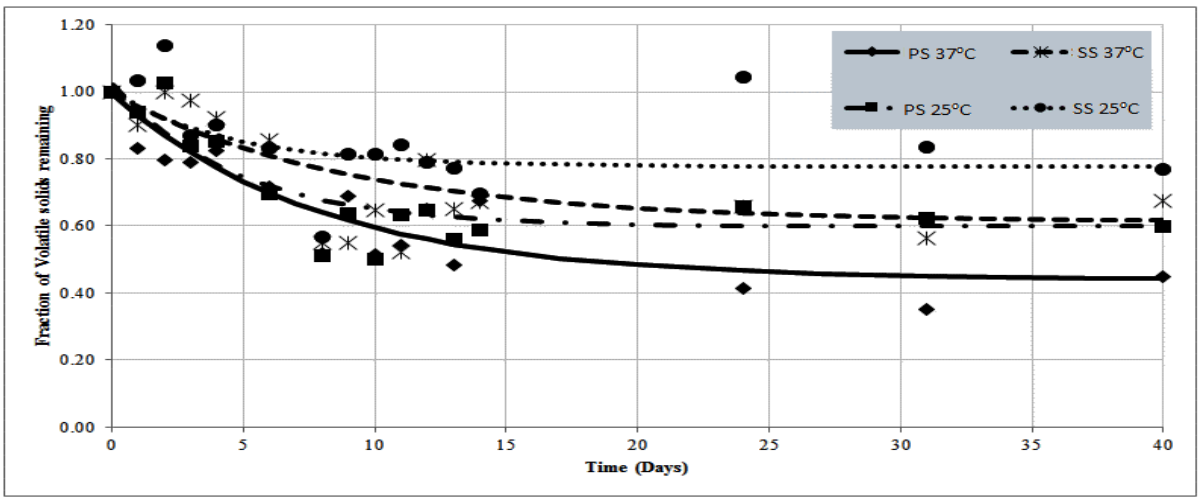


*Figure 1: Retained solid substrates corresponding to length (days) of experiment.*

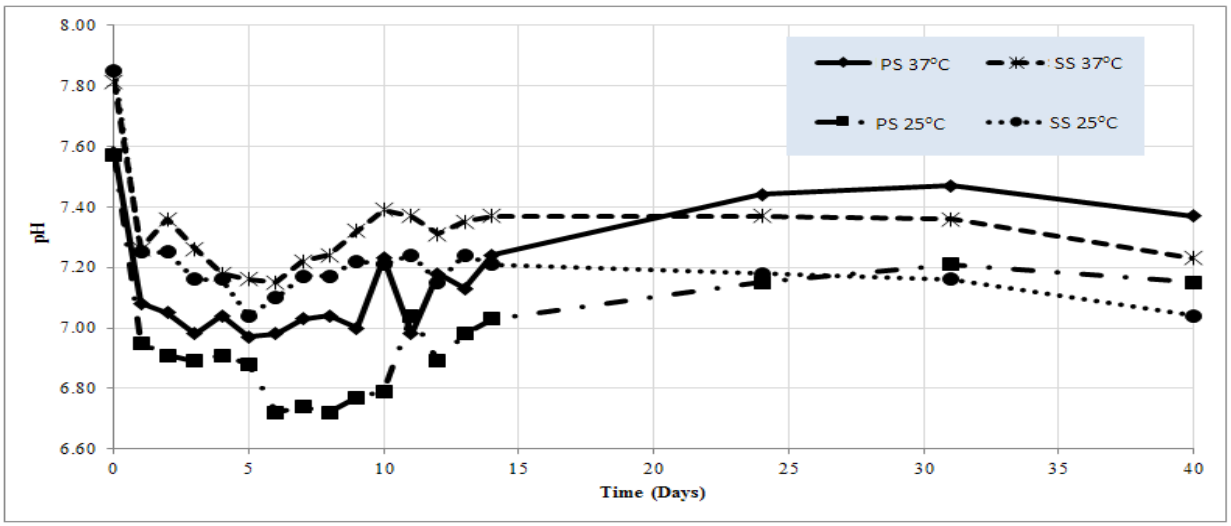


*Figure 2: pH values corresponding to length (days) of experiment.*

*Table 3: Hydrolysis rate constants ($k_h$) and statistical analysis*

| Substrate | Temp. (°C) | Fraction reduced (observed) | Fraction reduced (modelled) | Data points | $k_h$ ($d^{-1}$) | Bounds (±) | $R^2$ | RMSE | SSE |
|---|---|---|---|---|---|---|---|---|---|
| Primary sludge | 25 | 0.40 | 0.40 | 13 | 0.2089 | 0.0971 | 0.9325 | 0.0411 | 0.0169 |
| | 37 | 0.55 | 0.56 | 13 | 0.1278 | 0.0964 | 0.8103 | 0.8100 | 0.0656 |
| Secondary sludge | 25 | 0.23 | 0.22 | 12 | 0.2194 | 0.2094 | 0.7490 | 0.0420 | 0.0158 |
| | 37 | 0.33 | 0.38 | 11 | 0.1098 | 0.1036 | 0.8301 | 0.6570 | 0.0345 |

Initially, there were 18 data points for the tests and at least three outlying points, data outside 95% confidence level, had to be removed (Table 3) before a data fit was achieved. The secondary sludge tests had at least 6 outlying data points removed before a fit to the model was observed, but primary sludge tests had no more than five data points removed. These observed outlying data points are probably due to errors as a result of the sampling process, and the statistical error analysis (RSME and SSE in Table 3) provides additional details on the distribution of the observed data points relative to the model with respect to time [26].

The highest RSME value in Table 3 was for the primary sludge test at 37°C, observed with RSME = 0.8100, and also for the secondary sludge test at 37°C, observed with RSME = 0.6570. The differences between the predicted model values and the observed values for the other tests were small, as reflected by the small RSME values (Table 3), indicating that most of the observed values are close to the predicted model values. The SSE values provide another basis for comparison of the deviation of the predicted values from the observed values, and low SSE values in Table 3 indicate that the model, based on the hydrolysis rate and time, is predicting values that are close to the observed values. The hydrolysis rates constants ($k_h$) obtained (Table 3), are within the range of values observed in literature for primary sludge and secondary sludge, and summarized in Table 4.

Aldin [16] reported hydrolysis rate constants ($k_h$) for wastewater sludge within the range of 0.0096 to 1.94 $day^{-1}$ for primary sludge, 0.005 to 0.2 $day^{-1}$ for sewage sludge, with 0.08 to 2.0 $day^{-1}$ as the general range for most types of sludge. Eastman and Ferguson [35], Batstone *et al.* [36] and Siegrist *et al.* [37], reported hydrolysis rate constants for primary sludge between 0.2 - 0.5 $day^{-1}$ at mesophilic conditions, while Mahmoud [34] reported 0.23 $day^{-1}$ for settle-able solids from domestic wastewater at 35°C. Kassab *et al.* [33] reported hydrolysis rate constants based on first order kinetics as 0.006 $day^{-1}$ for seeded domestic wastewater sludge, and 0.004 $day^{-1}$ for unseeded domestic wastewater sludge.

From Table 4, the $k_h$ values reported by Aldin [16] were for a wide range of experiments, while Kassab *et al.* [33] reported the potential influence of high concentrations of detergents in their substrate as the reason for the low rate constants. Nielsen [38], Lee Ferguson and Brown well [39] and Jimenez-Gonzalez *et al.* [40], have reported poor anaerobic degradation due to detergents, mainly as a result of process inhibition [41- 42]. Even though the $k_h$ values from this study are close to the values reported by Mahmoud [34] and Luo *et al.* [15], and the values also fall within the ranges reported by Aldin [16], there is need for caution in comparison of the values due to the different experimental conditions. The summary presented in Table 4 indicates wide ranges of values for the hydrolysis rates, and this could be attributed to the differences in the nature and characteristics of the substrates and the experimental conditions. However, the correlation of the first order hydrolysis model, after discarding of data outliers, provides a basis for comparison of hydrolysis experiments with different substrates.

## 4. CONCLUSION

The potential for anaerobic reduction of domestic wastewater sludge at 25°C was evaluated and compared against anaerobic reduction at 37°C, and generally the secondary sludge (SS) showed lower reduction than the primary sludge. The results also revealed higher reduction at 37°C than at 25°C for the primary sludgetests, where over 55% of the volatile solids were reduced at 37°C, while only 40% reduction was observed at 25°C. For the secondary sludge (SS) test, over 33% of the volatile solids were reduced at 37°C, while only 22% reduction was observed at 25°C.

*Table 4: Summary of $k_h$ values from literature and this study*

| Study | Substrate | kh (day-1) | Conditions |
|---|---|---|---|
| Aldin[16] | Sewage sludge | 0.0050 – 0.2000 | Varying |
| Aldin[16] | Sludge | 0.0800 – 2.0000 | Varying |
| Aldin[16] | Primary sludge - lipids and proteins | 0.0096 – 0.1700 | Varying |
| Aldin[16] | Primary sludge - carbohydrates | 0.2100 – 1.9400 | Varying |
| Kassab et al. [33] | Domestic wastewater - sludge | 0.0060 | 25°C - seeded |
| Kassab et al. [33] | Domestic wastewater - sludge | 0.0040 | 25°C - unseeded |
| Mahmoud [34] | Domestic wastewater – Settle-able solids | 0.2300 | 35°C |
| This study | Primary sludge - volatile solids | $0.2089 \pm 0.0971$ | 25°C - seeded |
| This study | Primary sludge - volatile solids | $0.1278 \pm 0.0964$ | 37°C - seeded |
| This study | Secondary sludge - volatile solids | $0.2194 \pm 0.2094$ | 25°C - seeded |
| This study | Secondary sludge - volatile solids | $0.1098 \pm 0.1036$ | 37°C - seeded |

The rates of hydrolysis observed were higher at 25°C than at 37°C, while lower degrees of hydrolysis at 25°C than at 37°C were observed. $R^2$ values calculated based on the data from the reduction of the domestic wastewater sludge, indicated a good correlation of the hydrolysis model, digestion time and the reduction of the solids. Errors in the data indicated by data outliers, potentially due to the sample collection method adopted, constrained the curve fitting process and resulted in the discarding of data points, especially for the secondary sludge. Research into modification of the methodology, specifically the development of a reliable method to monitor the reduction of solids, may enhance the reliability of data collection, and lead to improvements in the hydrolysis process.

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