

Heat treatment and low pressure oxidation of aqueous sewage sludge

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HEAT TREATMENT AND LOW PRESSURE OXIDATION OF AQUEOUS SEWAGE SLUDGE

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Abstract—The subject of study was the effect of heat treatment and low pressure oxidation on:
—the specific filtration resistance,
—the COD, N and P content of the suspension.

Residence time distributions of the industrial oxidation reactor show considerable axial mixing of the continuous liquid phase and a high gas fraction (30%), which is probably caused by the existence of a foam layer.

Test runs at 165 and 185°C were carried out to compare specific filtration resistance of the treated sludge and the steam consumption of a heat treatment (Zimpro) unit at these two temperatures. Specific filtration resistance of the sludge treated at 165°C was 8 times higher than that of the treatment at 185°C. Moreover 20% more steam was consumed at the lower temperature. COD reduction of the sludge was in good agreement with the theoretical model.

INTRODUCTION

The heat treatment and low pressure oxidation of sewage sludge have been covered extensively in the literature. However, some process engineering aspects have not been studied. The purpose of the investigations carried out at Apeldoorn was to cover some of these aspects. Apeldoorn sludge is of domestic and industrial origin. It has 50% of inorganics and 85% of particles smaller than 60 μm . The composition of the fresh sludge is given in Table 1. The sludge contained 60% primary and 40% activated sludge. Since June 1969, this sludge has been treated in a heat treatment (Zimpro) unit, having a design capacity of 10.5 tons of dry solids per day. A second analogous Zimpro unit was put into operation early 1974. The flow sheet and description of this unit is shown in Fig. 1.

Wet-air oxidation of sludge

Laboratory scale apparatus and experimental procedures. Laboratory scale experiments were carried out to establish the influence of residence time, temperature and partial oxygen pressure on the heat treatment and the wet-air oxidation of fresh Apeldoorn sludge. A diagrammatic representation of the apparatus used is given in Fig. 2. The reactor consists of a stainless steel autoclave 1 with a liquid content of about 600 ml.

Mixing in the reactor is achieved by means of an electromagnetic "shaker" 2 with a frequency of 143 min^{-1} . A contact thermometer, connected to an iron-constantin thermocouple, keeps the temperature of the electrically heated reactor constant within 3°C.

Air is fed from a gas cylinder 5 via a reducing valve 6. The pressure and the gasflow are controlled by a sensitive reducing valve in the spent gas outlet and are measured with a manometer 8 and a flow meter

9 respectively. Droplets are separated from the spent gas stream and the gas is further dried in an absorber 11 filled with silicagel. To prevent evaporation losses as much as possible, the spent gas is cooled. The water formed on cooling flows back into the reactor. The oxygen concentration in the spent gas is measured with a Servomex oxygen measuring device 13.

Using an injection vessel 15, the sample to be studied is brought into the reactor, which is preheated to 20°C above the desired temperature. A dip-pipe and cooler 16 allow sludge samples to be taken from the reactor.

The process conditions of the Zimpro plant at Apeldoorn are: T (temperature): 180°C; P (total pressure): 26 atm; τ (mean residence time): 1 h. These conditions constitute the basis for the choice of the process conditions that were studied on a laboratory scale.

The temperatures used in the laboratory studies were 160, 180 and 200°C. The residence time at each temperature was 0.5, 1 and 2 h. The pressure was kept constant at 26 atm. Therefore the oxygen pressure at each temperature was different. The gas flow rate was 3.6 Nl h^{-1} . Samples were analysed for solid concentration and organic content specific filtration resistance, COD, total N, ammonia N and P. The methods of analysis were "Standard Methods" (1962).

Results of laboratory experiments

Dry solids concentration and organics content, COD measurements. Measurements of dry solids concentration and organics content of the dry solids during wet-air oxidation are given in Figs. 3 and 4. Dry solids dissolve in the course of a wet-air oxidation treatment, mainly by hydrolysis of the proteins and

Table 1. Fresh sludge composition (mean values, June 1972)

Dry solids %	Total COD kg m ⁻³	Total N g m ⁻³	Total P g m ⁻³	NH ₄ ⁺ -N g m ⁻³	Cu mg kg ⁻¹ dry solids	Cr mg kg ⁻¹ dry solids	Zn mg kg ⁻¹ dry solids	Hg mg kg ⁻¹ dry solids
9.2	72	1480	1370	57	200	457	520	2.7

carbohydrates present. The measurements given in Figs. 3 and 4 correspond with the occurrence of dissolution. The results indicate that the rate of hydrolysis increases as temperature increases.

Since the absolute quantity of ash in each sample is constant, any reduction in dry solids concentration that occurs is caused by the dissolution of organic material (Fig. 4). The dissolved compounds are oxidised by the air-oxygen present. The hydrolysis and oxidation reactions together determine the COD concentration in the suspension as a function of time. Figures 5 and 6 show the experimental relations for filtrate COD as well as total suspension COD.

Figure 5 shows that at 160°C, filtrate COD increases as the residence time increases, i.e. the hydrolysis rate is always greater than the oxidation rate. At 180–200°C, however, oxidation becomes faster than hydrolysis after 0.5 or 1 h, which gives rise to a maximum in the curve.

To produce a filtrate with low COD-concentration one can either use a low temperature and a short residence time or use a high temperature and a long residence time. Figure 6, which represents the COD of the suspension as a function of time, also shows an appreciable increase in the oxidation rate with temperature. These measurements fit the theoretical model of Ploos van Amstel (1971; 1972). He concludes that raw sludge consists of three groups of material as to oxidisability—65% reactive material A—25% less reactive material B—10% non-reactive material C. Furthermore, it is apparent from his investigations that the oxidation rate can be described

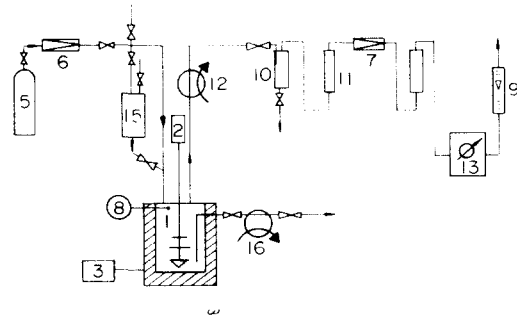


Fig. 2. Flow diagram laboratory apparatus for wet air-oxidation.

as a first-order reaction with respect to organic material as well as oxygen for the two groups A and B, each having its own reaction constant. Since the oxygen concentration was not known, it was calculated from the measured oxygen concentration in the spent gas.

The solid curves in Fig. 6 represent the results of these calculations. The values for the reaction rate constants k_a and k_b used are those given by Ploos van Amstel (1971, 1972). The agreement between theory and experiments is very good.

Specific resistance

As has been discussed earlier, heat treatment and wet-air oxidation of sludge aim at conditioning it to render it more readily dewaterable. The specific resistance is widely used as a characteristic measure of the

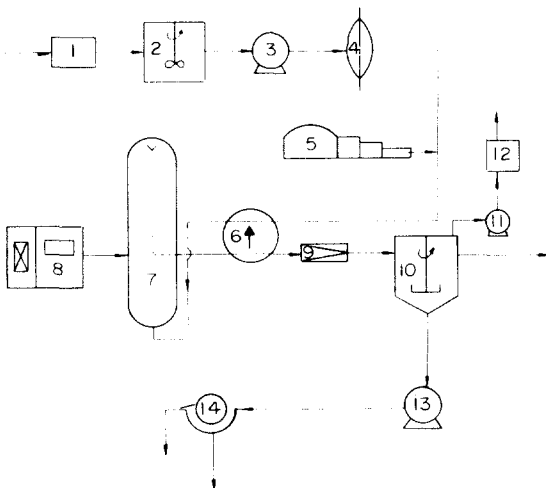


Fig. 1. Flow diagram Zimpro unit at Apeldoorn.

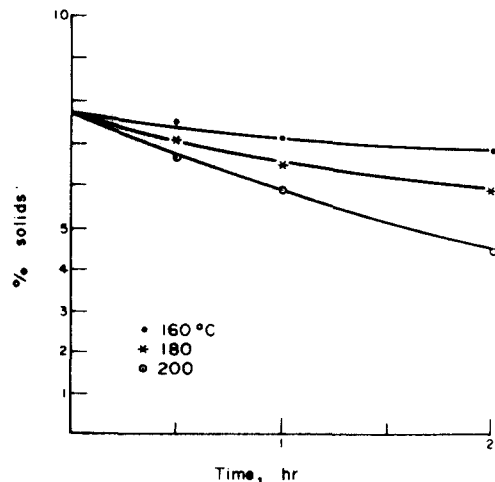


Fig. 3. Dry-solids content of the sludge-suspension during thermal-oxidative treatment.

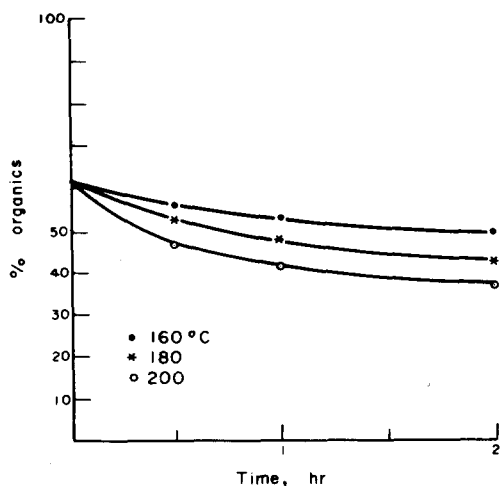


Fig. 4. Organics-content of the dry solids during thermal-oxidative treatment.

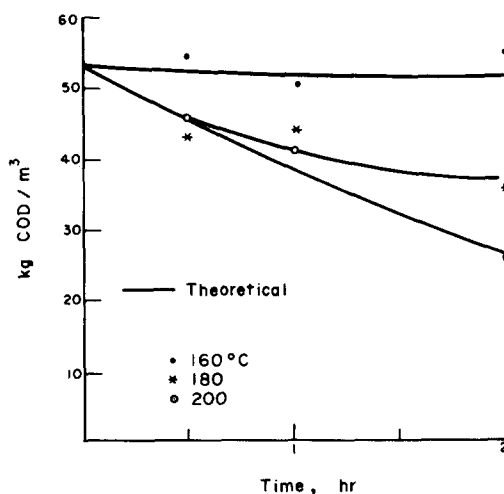


Fig. 6. Suspension COD vs time during thermal-oxidative treatment.

ease of dewatering. Brooks (1970) and Heusler (1969) give a value of $40 \times 10^7 \text{ s}^2 \text{ g}^{-1}$ as a criterion of filterability using an industrial vacuum filter. Although in the literature several definitions of the specific resistance are used, this quantity for a non-compressible filtercake will here be defined after e.g. Swanwick (1961) and Coulson and Richardson (1959). The apparatus of Swanwick was used.

Although this investigation (Crujlsberg and Jakma, 1973) led to the conclusion that fresh as well as oxidised sludge form a somewhat compressible filtercake, it is possible, by standardising the filtration pressure, to define the specific resistance in the same way as above (in our experiments the filtration pressure was standardised at 160 mm Hg). A straight filtration curve was not obtained in all cases (see for instance Fig. 7, 200°C, 0.5 h), due to clogging of the filtercake, which increases the specific resistance. This increase may be neglected. Table 2 shows the measurements for sludge treated by wet-air oxidation.

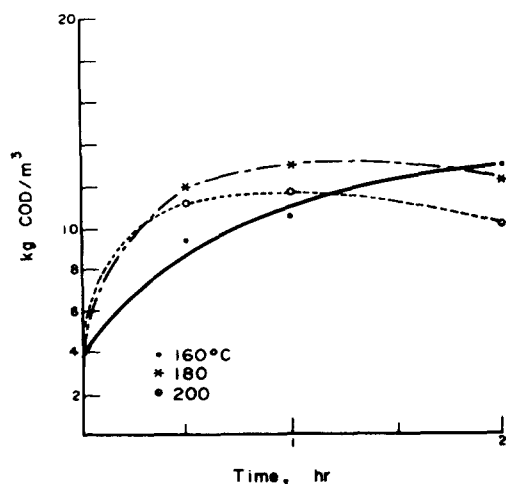


Fig. 5. Filtrate COD vs time during thermal-oxidative treatment.

The table also shows the specific resistances of fresh sludge and chemically conditioned sludge (with FeCl_3 and lime). Non-treated sludge obviously has poor filtration characteristics. The values were greater than those given by Brooks (1970) and Heusler (1969). The sludge has strong waterbinding properties, which are overcome in the wet-air oxidation treatment. Further it is apparent from the table that in general the resistance increases as the oxidative treatment proceeds. The cause of this is the formation of very small particles, which in course of time clog the filter.

N and P analyses

The fact that sludge, treated by wet-air oxidation might be used as soil-conditioning material, makes it important to know what happens to the nitrogen and phosphorus in the course of the process. The results for N and P concentration are given in Table 3.

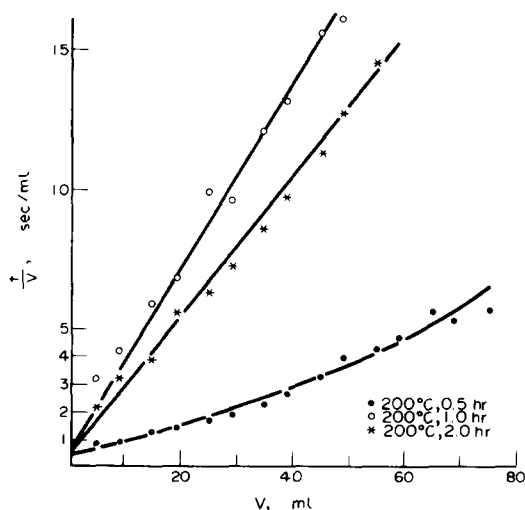


Fig. 7. Measurement of filtration-resistance of thermal-oxidative treated sludge (laboratory scale). Filtration pressure $\Delta p = 160 \text{ mm Hg}$.

Table 2. Specific resistance of thermal-oxidative treated sludge

Temperature	160°			180°			200°		
Residence time (h)	0.5	1	2	0.5	1	2	0.5	1	2
Specific filtration resistance ($10^7 \text{ s}^2 \text{ g}^{-1}$)	6.2	5.5	5.0	1.6	5.1	5.2	1.7	9.2	8.9
Filtration pressure: 160 mm Hg									
Specific resistance of fresh sludge	$75 \times 10^7 \text{ s}^2 \text{ g}^{-1}$								
Specific resistance of chemically treated sludge	$11 \times 10^7 \text{ s}^2 \text{ g}^{-1}$								

Table 3. P and N concentrations

	fresh sludge	160°C			180°C			200°C		
		0.5	1	2 h	0.5	1	2 h	0.5	1	2 h
P in dry solids ($\text{g kg}^{-1} \text{ d.s.}$)	0.6	1.4	1.8	2.3	2.2	1.9	2.2	1.6	0.7	1.2
P in filtrate (g m^{-3})	38	50	43	37	37	51	63	40	49	47
Total N in dry solids ($\text{g kg}^{-1} \text{ d.s.}$)	13.3	11.2			9.2					

The results indicate that phosphorus is hardly dissolved in the wet-air oxidation process. The total N content of the solids decreases slowly (Table 3). This is also apparent from Figs. 8 and 9, where the ammonia N and total N concentration in the filtrate are given. The total quantity of nitrogen decreases with increasing temperature and residence time. This is explained by the fact that NH_3 and N containing organic compounds formed as a result of the hydrolysis and oxidation reactions, are stripped from the liquid.

Full-scale measurements

The residence time distribution in the reactor. Although the industrial oxidation reactor has a large L/D ratio one cannot conclude from this that the

liquid phase moves through the reactor in plug flow. The residence time distribution of the liquid water-phase in the reactor was measured by introducing a pulse injection of 18 kg of NaCl solved in 60 l of water over a period of about 1 min. Concentrations were determined by conductivity measurements.

Because of the local temperature and pressure the pulse injections had to be made at the inlet of pump no. 3 (see Fig. 1). The pulse response of the reactor can then only be determined if it is known how the pulse was influenced by the pumps and heat-exchanger at the time it arrived at the reactor inlet. By measuring the response at the reactor outlet also, the residence time distribution in the reactor can then be determined. Figure 10 shows the response measured at the reactor inlet. Further analysis of this

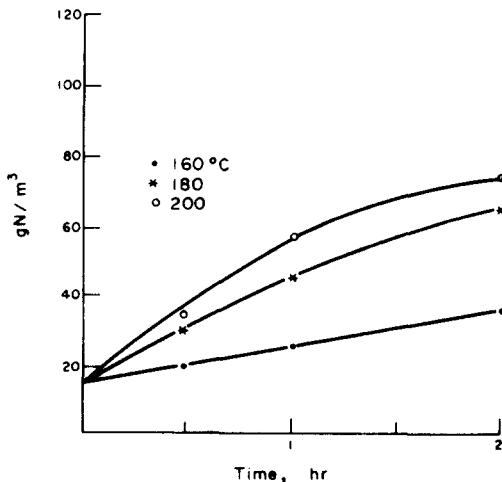
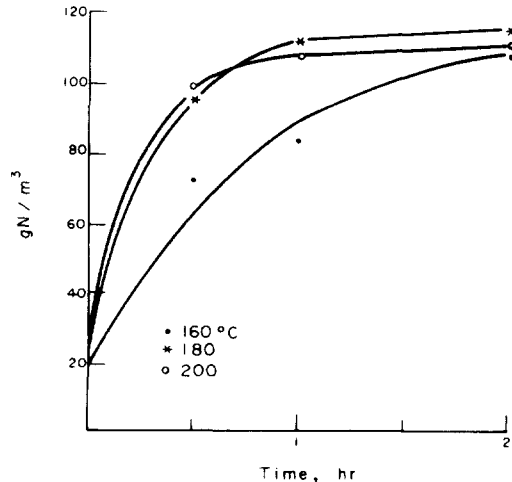
Fig. 8. Filtrate $\text{NH}_3\text{-N}$ concentration.

Fig. 9. Filtrate total N concentration.

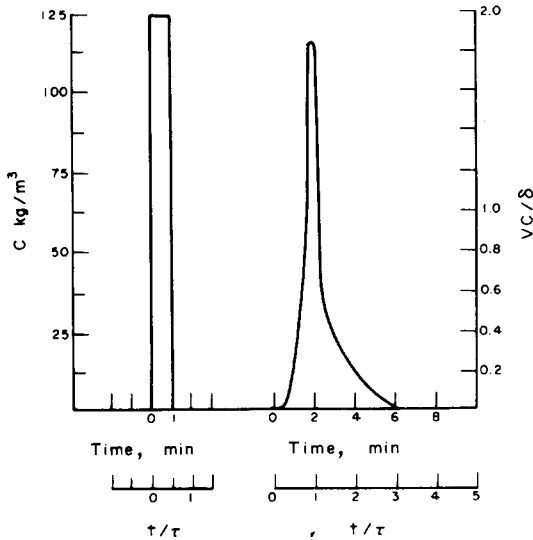


Fig. 10. Influence of the heat exchanger on the pulse injection.

response curve gives a mean residence time in the heat-exchanger and pipes of 2 min. With an observed liquid flow-rate of $8.4 \text{ m}^3 \text{ h}^{-1}$ it follows that the volume occupied by the liquid phase in this system is 0.28 m^3 . The standardised C diagram using these Figures is also given in Fig. 10 (Danckwerts, 1953).

From this standardised curve it follows that the increase in standard deviation σ/μ compared with the original pulse is so small that it is justifiable still to consider the response at the reactor inlet as a pulse injection. The response at the reactor outlet has been measured twice. From these results the response of the reactor to a step-function as input has been calculated by numerical integration. This so-called cumulative residence time distribution or F-curve is shown

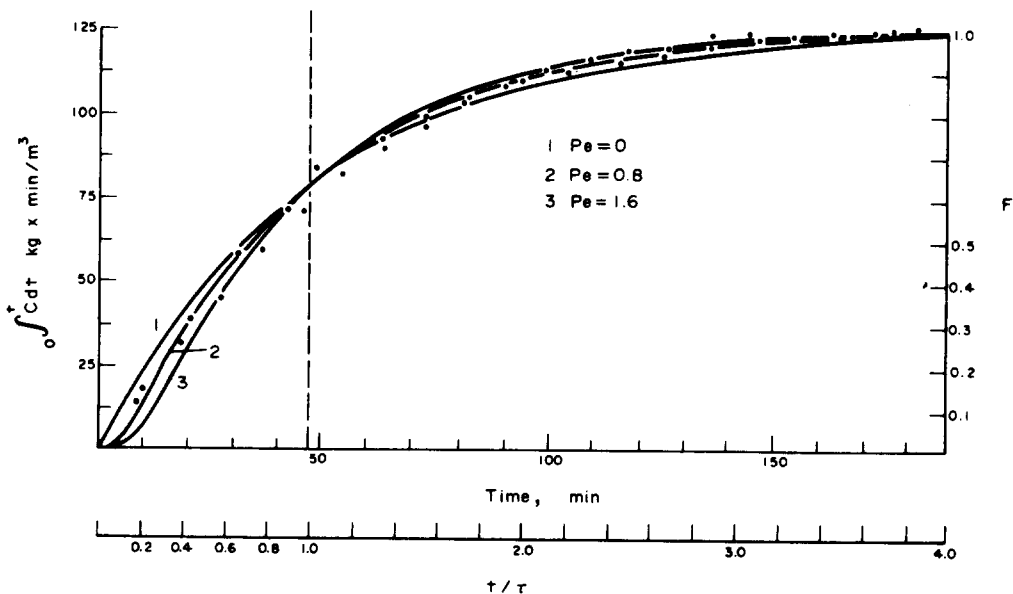


Fig. 11. Cumulative residence time distribution in the reactor.

in Fig. 11. The mean residence time calculated from this curve is 48.5 min.

In Fig. 11 the observed response is compared with the "continuous backflow model" for several values of the Péclet number. The theoretical curves were numerically calculated by Brenner (1962). From Fig. 11 it is apparent that the measurements are close to the curves with low Péclet numbers ($Pé = 1$ to 2) i.e., appreciable axial mixing occurs, caused by strong circulation of liquid in the reactor, which in turn might be occasioned by the way in which the air is dispersed in the reactor. Together with the sludge the air is introduced through a wide pipe (diameter 5 cm) in the bottom of the reactor. Because of this construction big air bubbles will rise rapidly in the centre of the column, causing a circulation of liquid as shown diagrammatically in Fig. 12. With respect to the mixing one can conclude that for practical purposes it is acceptable to consider the reactor as an ideal mixer.

The volume occupied by liquid in the reactor can be calculated from $V_L = Q_s \cdot \tau_L$. From this it follows that $V_L = 6.8 \text{ m}^3$. While the reactor volume $V_r = 10 \text{ m}^3$, the gas fraction β is calculated as

$$\beta = (V_r - V_L)/V_r = 0.32.$$

This high value is probably due to foam formation in the reactor.

The oxidation rate

The COD-conversion in the Zimpro reactor is determined by the kinetics of the oxidation reactions and the mixing in the reactor. If data about these quantities are known, the COD conversion can be calculated. This conversion has been experimentally determined from: the oxygen content of the spent gas; COD difference between fresh and oxidised sludge.

The oxygen concentrations in the spent gas have been measured by a Beckman Oxygen Analyser (model 777) and an Orsat Unit. The oxygen concentration in the spent gas is rather constant. On three separate days average values of this concentration of 14.8, 14.4 and 14.8% were found. From the mean oxygen concentration in the spent gas (14.7%) and the normally used air flow rate ($270 \text{ Nm}^3 \text{ h}^{-1}$) it follows that the oxygen consumption rate in the reactor is 24.4 kg h^{-1} . The COD concentration of the influent strongly fluctuates. Using the mean value of 55 kg COD m^3 together with a sludge flow rate of $8.4 \text{ m}^3 \text{ h}^{-1}$ the mean COD loading is calculated to be $462 \text{ kg COD h}^{-1}$. On the basis of the oxygen uptake a conversion of 5.3% is calculated.

Theoretically it follows from the kinetic model given that the COD conversion rate R in the ideally mixed reactor is given by:

$$R = R_a + R_b = k_a \cdot V_l \cdot A_e \cdot O + k_b \cdot V_l \cdot B_e \cdot O$$

where $V_l = (1 - \beta)V_r$.

In this reaction A_e and B_e are the COD concentrations in the reactor of the reactive and the less reactive components of the sludge respectively. At 185°C the values of the reaction rate constants k_a and k_b are 3.8 and $0.4 \text{ m}^3 \text{ kg}^{-1} \text{ h}^{-1}$ respectively (Ploos van Amstel, 1971). The COD of the sludge fed to the reactor varies strongly, the mean value being 55 kg m^3 . For the oxygen concentration of the liquid phase in the reactor a mean value of $0.027 \text{ kg O}_2 \text{ m}^{-3}$ ($\pm 10\%$) has been observed. Using these figures and the values given before, a conversion rate of $R = 24 \text{ kg COD h}^{-1}$ is calculated. This results in a conversion of 5.2%. Although this theoretical value is in good agreement with the conversion calculated on the basis of the oxygen uptake, the COD reduction observed is generally higher 10 to 15%. The difference is probably caused by the stripping of volatile components from

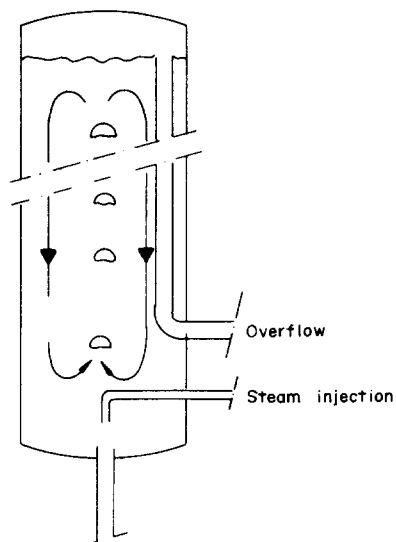


Fig. 12. Circulation of liquid in the reactor.

Table 4. Spent gas analysis

Component	Concentration mg m^{-3}
Ketone	6500
Formaldehyde	20
Hydrocarbons	1300
Alkenes	1000
CO	1400
H ₂ S	0
SO ₂	0
Mercaptan	0
NH ₃	0
Alcohols	0
NO/NO ₂	0

the liquid, e.g. carboxylic acids, aldehydes and ketones.

Using Dräger tubes, it was hoped to get an impression of the concentration of some components in the spent gas. A full analysis, however, could not be made. The results are given in Table 4. Because of the high organic content of the gas it is treated in a catalytic afterburner.

Influence of temperature on performance

A test run has been carried out at 165°C instead of at 185°C . The energy balance of the reactor is determined by three quantities: the reaction heat liberated, the heat necessary to bring the sludge/air mixture to the temperature in the reactor, and the heat losses. From this the quantity of heat required as steam can be determined. To compare the steam quantities to be supplied at 165 and 185°C respectively, it is assumed that the heat losses at both temperatures are equal. From temperature measurements at the outlet of the heat-exchanger the heat necessary for heating the sludge/air mixture that comes out of the exchanger to reaction temperature, is almost the same in both cases and equal to $307,000 \text{ kcal h}^{-1}$. From the heat of combustion of 3.18 kcal g^{-1} COD given by Zimmermann and Teletzke (1958, 1964) the reaction rate at which heat is liberated in the reactor can be calculated to be $32,000 \text{ kcal h}^{-1}$ at 165°C and, as a result of the higher reaction rate, $77,500 \text{ kcal h}^{-1}$ at 185°C . At 165°C $275,000 \text{ kcal h}^{-1}$ and at 185°C $230,000 \text{ kcal h}^{-1}$ will have to be supplied to the reactor as steam. Therefore it is uneconomical to lower the temperature from 185°C to 165°C , as at 165°C about 20% more steam is consumed. During this test run the specific resistance of the treated sludge was also measured (Table 5).

The mean value was $60 \times 10^7 \text{ s}^2 \text{ g}^{-1}$ at 165°C , while sludge treated at 185°C had a mean filtration resistance of $8 \times 10^7 \text{ s}^2 \text{ g}^{-1}$. Thus the rise in specific

Table 5. Specific resistances of oxidised sludge

Temperature	Specific filtration resistance ($10^7 \text{ s}^2 \text{ g}^{-1}$)			
165 C	75	50	64	50
185 C	8	11	4	7

Table 6. Specific filtration resistance of chemically treated sludge

Temperature	150°C		160°C		180°C		200°C		220°C					
Residence time (h)	0.5	1	0.25	0.50	1	0.50	1	2	0.25	0.50	1	0.50	1	2
Specific resistance r ($10^7 \text{ s}^2 \text{ g}^{-1}$)	67	18.5	4.30	5.60	1.65	2	0.79	0.07	0.74	0.07	0.09	0.04	0.04	0.04

resistance does not favour lowering the temperature to 165°C. On the basis of batch experiments (Table 2) one would not expect this decrease in temperature to have a large influence on the specific resistance resulting from a continuous process. Such extrapolation is not justifiable, however, because the influence of the axial mixing of the liquid phase in the reactor on the specific resistance of the end-product is then overlooked. Axial mixing has two effects:

1. That part of the sludge having a small residence time will still have rather high specific resistance, causing the resistance of the end-product to rise. This effect will be greater at lower than at higher temperatures (for comparison see the measurements in Table 2 for $\tau = 1/2 \text{ h}$ at 160, 180 and 200°C).

2. The portion of the sludge staying too long in the reactor also causes an increase in specific resistance of the end product because of the formation of very small particles. This effect also causes clogging of the filtercake as mentioned earlier.

Although these effects are not quantifiable, it can be concluded from the measurements that axial mixing has, especially at lower temperatures, an unfavourable influence on the specific resistance of the treated sludge. The expectation is that at lower temperatures a decrease in axial mixing will have a favourable effect on the specific resistance.

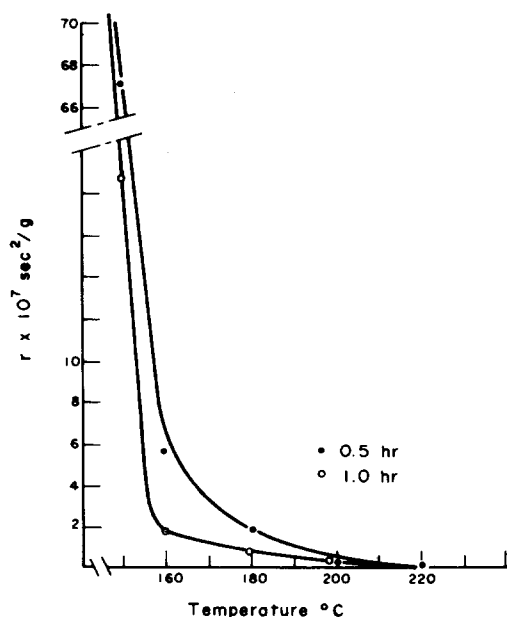


Fig. 13. Filtration resistance of thermal treated sludge.

Heat treatment of sludge

Filtration resistance. The heat treatment experiments were performed in the same apparatus as the wet-air oxidation experiments described earlier. Instead of a continuous gasflow, the system was now pressurised with nitrogen at the start of an experiment.

Because these experiments were carried out with sludge coming from a new sewage treatment plant, the results obtained are not directly comparable with those from the wet-air oxidation experiments.

Notable in these experiments was the "sticky" behaviour of the sludge, after being heat-treated for some time (*ca.* 1 h). Sticking of solids to the reactor wall occurred. The filtration resistance measured as a function of time and temperature is given in Table 6. The filtration resistance decreases at increasing temperature and residence time. After a certain residence time depending on the temperature, however, further treatment no longer resulted in appreciable improvement. After a constant residence time, the filtration resistance decreases sharply, if the temperature varies from 150 to 160°C; treatment at temperatures above 160°C gives far less improvement (Fig. 13). The resistances found are almost all lower than the Figure given by Brooks and Heusler. The conclusion is that heat treatment does not result in a higher specific resistance than wet-air oxidation. Without proper measures being taken, it can be expected, however, that one can encounter problems in practice at residence times above 1 h because of the sludge sticking to the reactor wall.

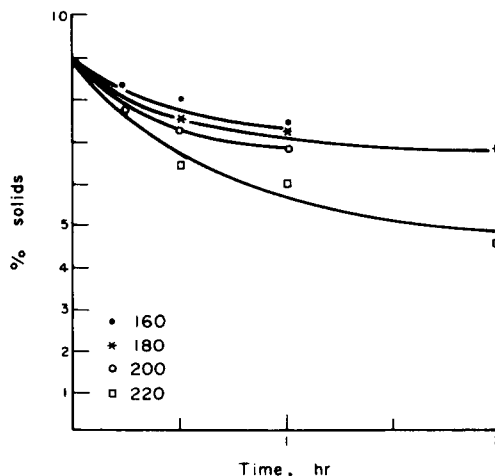


Fig. 14. Dry solids concentration of the sludge-suspension during thermal treatment.

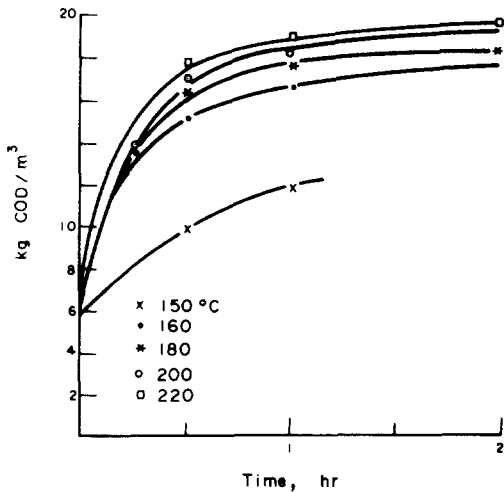


Fig. 15. Filtrate COD during thermal treatment.

Dry solids and COD after heat treatment

The solids content of the suspension and the COD-concentration of the filtrate during heat treatment are shown in Figs. 14 and 15. The filtrate COD approaches a constant value after 1 to 2 h. This arises from the fact that hydrolysis occurs without oxidation. The quantity of solids dissolved by hydrolysis increases with temperature. The hydrolysis rate is also temperature dependent. According to Ploos van Amstel (1971), this rate is due to the reactive group of solids present in the sludge, and may be described as an irreversible first-order reaction. The value for the hydrolysis rate constant calculated from Fig. 15 varies from 2.3 h^{-1} at 150°C up to 4.0 h^{-1} at 220°C . The values for Apeldoorn sludge are about a factor of 10 lower than those for Eindhoven sludge (1971). Besides the difference in the nature of these sludges, no explanation has been found of the difference in behaviour.

CONCLUSIONS

1. The hydrophylic properties of the sludge are overcome and the filtration characteristics considerably improved by heat treatment as well as wet-air oxidation. This is shown by a decrease of the specific resistance from $75 \times 10^7 \text{ s}^2 \text{ g}^{-1}$ for fresh sludge to values of about $1 \times 10^7 \text{ s}^2 \text{ g}^{-1}$ dependent on the length and temperature of treatment.

2. Although heat treatment does not give a higher specific filtration resistance than wet-air oxidation, the sludge becomes sticky after about 1 h. In practice this might cause problems at residence times greater than 1 h.

3. The phosphorus in the sludge solids does not dissolve appreciably during wet-air oxidation.

4. The total N in the solids decreases, depending upon the time and temperature of treatment.

5. Appreciable axial mixing occurs in the Zimpro reactor at Apeldoorn. At lower temperatures this adversely affects the specific filtration resistance of the sludge. A test-run of this reactor at 165°C produced a sludge with a specific resistance of $60 \times 10^7 \text{ s}^2 \text{ g}^{-1}$; at 185°C this resistance is about $8 \times 10^7 \text{ s}^2 \text{ g}^{-1}$. Furthermore, this lower temperature is less favourable since at 165°C about 20% more steam is needed than at 185°C .

6. The hold-up of gas in the system is high (ca. 30%). This is probably due to foam formation in the reactor.

7. The oxidation level measured from the oxygen uptake from the gas is 5.3%. This is in agreement with the theoretically calculated value. The observed conversion on COD-basis of the sludge is 10–15%. The difference is probably due to the stripping of volatile components.

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