

Original Article

Optimising Incineration Parameters of Wastewater Sludge Ash Production for a Sustainable Environment and Construction Materials

Kobe Samuel Mojapelo^{1*}, Everardt Andre Burger¹, Jacques Snyman¹, Idowu David Ibrahim², Charity Maepa³

¹Department of Civil Engineering, Tshwane University of Technology, Pretoria, South Africa.

²Department of Industrial Engineering, Operations Management and Mechanical Engineering, Vaal University of Technology, Vanderbijlpark, South Africa.

³Institute for Nano Engineering Research (INER) and Department of Chemical Metallurgy and Materials Engineering, Tshwane University of Technology, Pretoria, South Africa.

*Corresponding Author : MojapeloKS@tut.ac.za

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Abstract - South Africa produces approximately 639,000 dry tonnes of wastewater sludge annually, creating disposal challenges and opportunities for resource recovery through Wastewater Sludge Ash (WSA). This study optimised and interpreted the thermal conversion of Polokwane Wastewater Sludge (WWS) to WSA by evaluating atmosphere and heating rate-dependent decomposition, as well as kinetic and thermodynamic parameters. Thermogravimetric analyses were conducted to 900 °C at 10, 20 and 40 °C/min under air and nitrogen (N₂). Three decomposition stages were identified: moisture loss (25-150 °C), organic decomposition (200-500 °C) and carbonate decomposition (600-900 °C), producing approximately 70% WSA in air. Coats-Redfern analysis showed higher effective activation energies in air (118.6 and 142.1 kJ/mol) than in N₂ (94.8 and 107.5 kJ/mol), with strong linear fits ($R^2 = 0.986-0.994$). Decomposition was endothermic, with a higher ΔH in air, while positive ΔG indicated non-spontaneous thermal decomposition. XRD identified quartz, calcite and haematite, with increased crystallinity at 600 °C. FTIR confirmed organic burnout at 600 °C through the disappearance of C-H and C=O/N-H bands.

Keywords - Thermal optimisation, Incineration parameters, Wastewater sludge ash, Construction materials, Quality control.

1. Introduction

South Africa generates approximately 639,000 dry tonnes of Wastewater Sludge (WWS) annually, presenting substantial disposal challenges and environmental hazards [1]. Various alternatives addressing the escalating WWS production are documented in the literature, including thermal treatment [2, 3]. Controlled incineration is a promising technique for volume reduction and resource recovery, converting WWS into Wastewater Sludge Ash (WSA), which can serve as a potential supplemental cementitious material in construction.

The physical and chemical quality of WSA is strongly dependent on incineration temperature, environment, and residence time. Insufficient processing leads to inadequate removal of organic matter, low pozzolanic activity and poor-quality construction material. Therefore, rigorous parameter optimisation is needed to achieve uniform quality for structural applications [4, 5]. The use of WSA also has great environmental benefits, as it is in line with the circular economy and may reduce carbon emissions compared with

conventional cement production [6, 7].

There has been considerable research on the characterisation and management of WWS in the Limpopo Province of South Africa. Gantayat et al. [8] studied heavy metal concentrations in municipal sewage from various municipalities in Limpopo, providing important baseline data on sludge quality in the region. Montwedi et al. [9] studied the environmental effects of wastewater discharges in the Limpopo River basin, and Netshiongolwe et al. [10] analysed heavy metal contamination in the regional water distribution network. However, these basic environmental studies have not been followed by a thorough optimisation of thermal treatment settings for this regional sludge. However, there is a considerable information vacuum regarding optimised, systematic thermal treatment parameters for South African sludge characteristics. Systematic optimisation of the incineration process for energy efficiency and high-quality WSA production remains limited. Previous research has investigated the viability of using raw sludge and the fundamental thermal parameters of the produced WSA.



Recent developments in thermal treatment technology have highlighted the need for site-specific optimisation approaches. Specifically, the literature lacks extensive studies on the combined effects of temperature, atmosphere and residence time on the quality of Polokwane WSA. The absence of solid-state kinetic parameters for the thermal decomposition of WSA hampers the design of industrial processes, and there is no practical, accessible quality-control approach for municipal operators that does not require expensive analytical equipment. These inadequacies constitute a major impediment to the application of WSA in the South African construction industry. This study examines how atmospheric conditions, temperature and heating rate affect the transformation of Polokwane WWS into WSA. Its main contribution is to provide a process-level interpretation of raw TGA/DTG behaviour, stage-specific Coats-Redfern model fitting and effective thermodynamic parameters. Ash characterisation, strength and leaching results are integrated as linked validation of the application pathway, except where disclosed below. Related-work disclosure: This study is part of a wider Polokwane WSA research programme. Mojapelo et al. [11] report on the application-level mechanical, durability and chemical characterisation, and TCLP outcomes of WSA-concrete. The thermal-process analysis and the associated material and concrete evidence are treated as programme validation rather than as independent or newly discovered evidence in this work.

2. Materials and Methods

2.1. Material Collection and Preparation

WWS samples were collected from the Polokwane WWTP in accordance with the SANS 5667-13:2007 protocols. During each sampling session, three composite samples were collected from the drying beds. Fresh samples were processed within 24 hrs and dried at 105 °C for 24 hrs to reach a constant weight. This plant treats municipal and industrial wastewater using conventional activated sludge treatment. To avoid contact and inhalation, Personal Protective Equipment (PPE) and N95 respirators have been established as preventive measures.

2.2. Physical and Chemical Properties of Polokwane WWS

Testing was carried out according to South African National Standards (SANS) and American Society for Testing and Materials (ASTM) standard methods to ensure accurate and reproducible results. Three sets of testing were done. The dust content of the WWS was observed to be 10.6% (SANS 201:2008), and the moisture content was observed to be 5.2% (SANS 3001-GR20:2010). The material density was 1.23 g/cm³ (SANS 3001-AG23:2012) and the compacted bulk density 316 kg/m³ (SANS 5845:2006). The pH was 5.07 (TMH1, Method A.21). These are important parameters for assessing the suitability of materials for raw resource treatment in construction. The physical and chemical properties of the sample are listed in Table 1.

2.3. Thermal Analysis and Optimisation

Thermogravimetric Analysis (TGA) and Derivative Thermogravimetry (DTG) were carried out using a TA Instruments SDT Q600, as described in Table 2. Samples were weighed to 10±0.5 mg and heated from ambient temperature to 900 °C at various heating rates (10, 20, 40 °C/min) in air and nitrogen atmospheres, with a flow rate of 100 mL/min. The 40 °C/min rate was selected for kinetic analysis because it yielded well-resolved DTG peaks for Coats-Redfern regression. The 10 and 20 °C/min runs were used to assess the consistency of the thermal response across the tested heating rates. The TGA data shows three decomposition steps:

- Stage 1: moisture loss 25-150 °C, about 5% mass loss,
- Stage 2: organic decomposition 200-500 °C, about 21% mass loss,
- Stage 3: carbonate decomposition 600-900 °C, about 0.6% mass loss.

The total weight loss across these stages was 26.6%, and the inorganic residue and remaining compounds made up 73.4% of the original mass. In air, complete combustion of the sample resulted in a final mass of 70.4%, representing about 70% WSA after total thermal burnout.

Table 1. Physical and chemical properties of WWS from the Polokwane WWTP

Test	Sample results	Standard methods
Relative density	1.230	SANS 3001-AG23:2012
Compacted bulk density	316 kg/m ³	SANS 5845:2006
Dust content	10.6%	SANS 201:2008
Moisture content	5.2	SANS 3001-GR20:2010
pH	5.07	TMH1 Method A21

Table 2. Experimental design for thermal optimisation

Parameter	Range/Conditions	Rationale
Temperature Range	25-900 °C	Complete thermal profile
Heating Rate	10, 20, 40 °C/min	Rate comparison; 40 °C/min primary rate
Atmosphere	Air, Nitrogen	Oxidative vs. pyrolytic conditions
Sample Mass	10±0.5 mg	Analytical precision
Gas Flow Rate	100 mL/min	Standard conditions
Residence Time	35 minutes	Proposed for pilot-scale validation, not directly measured

The difference between the total inorganic residue (73.4%) and the final ash yield (70.4%) arises from volatile inorganic compounds such as water vapour and other oxides released during high-temperature combustion. The chosen condition and ash-yield interpretation is an extension of the previous article by the authors [11].

2.4. Thermodynamic Parameters

The thermodynamic parameters for the main decomposition stages of Polokwane WWS were calculated under air and nitrogen atmospheres. The change in enthalpy (ΔH) of decomposition was calculated to be endothermic. Air ΔH for stages 2 and 3 were 113.3 and 133.9 kJ·mol⁻¹, respectively, which were higher than those for nitrogen (89.7 and 99.3 kJ·mol⁻¹). This indicates that oxidative burning and carbonate decomposition in an oxygen-rich environment require more energy.

The ΔS of nitrogen was remarkably negative (-149.6 J·mol⁻¹ K⁻¹) for stage 2 due to a more orderly decomposition process. The Gibbs free energy (ΔG) of formation for the decomposing complexes is positive at all stages. The ΔG for nitrogen at Stage 3 is 227.1 kJ·mol⁻¹, which is significantly lower than that of air (232.7 kJ·mol⁻¹). This suggests that pyrolysis decomposition is more thermodynamically favourable in the absence of oxygen.

In the Vorhees theory, the negative entropy change implies that decomposition occurs in the solid state. The ΔS of nitrogen in stage 2 was highly negative (-149.6 J·mol⁻¹ K⁻¹), indicating that the decomposition process is more orderly. The results indicate a lower overall energy activation but a larger thermodynamic barrier for nitrogen. Thus, thermodynamically, nitrogen is less reactive than air.

Therefore, nitrogen's reactivity is less favourable in thermodynamic terms compared to air. The thermodynamic parameters were evaluated using Equations 1, 2, and 3 based on transition state theory:

$$\text{Change in enthalpy } (\Delta H) \\ \Delta H = E_a - R \cdot T_m \quad (1)$$

$$\text{Gibbs free energy change } (\Delta G): \\ \Delta G = E_a + R \cdot T_m \cdot \ln(kB \cdot T_m - h \cdot A) \quad (2)$$

$$\text{Entropy Change } (\Delta S): \\ \Delta S = (\Delta H - \Delta G) / T_m \quad (3)$$

Where:

E_a : Activation energy (obtained from kinetic analysis),
 R : The universal gas constant (8.314 J·mol⁻¹K⁻¹),
 T_m : The peak temperature in Kelvin (K) from the DTG curve,
 A : The pre-exponential factor from the Arrhenius equation,
 kB : The Boltzmann constant (1.381×10^{-23} J·K⁻¹),
 h : The Planck constant (6.626×10^{-34} J·s).

2.5. Kinetic and Thermodynamic Analysis

The kinetic parameters for thermal decomposition were determined using the Coats-Redfern integral method, a well-established technique for analysing solid-state reactions. This method assumes that the reaction occurs in a single step with constant activation energy, thereby simplifying complex processes such as sludge decomposition. Consequently, the kinetic parameters should be regarded as effective values more suitable for process design than fundamental mechanistic coefficients. Equation 4 can be used for this method. Multiple kinetic models were tested to identify the best-fit mechanism for each decomposition stage.

$$\ln \left[\frac{g(\alpha)}{T^2} \right] = \ln \left(\frac{AR}{\beta E_a} \right) - \frac{E_a}{RT} \quad (4) \\ \alpha = \frac{m_0 - m_t}{m_0 - m_f}$$

Where:

α : is the fractional conversion,
 m_0 is the initial mass,
 m_t is the mass at time t (corresponding temperature),
 m_f is the final mass after completion of the decomposition stage.
 $g(\alpha)$: integral form of kinetic model,
 T : absolute temperature (K),
 A : pre-exponential factor (min⁻¹),
 R : universal gas constant (8.314 J/mol·K),
 β : heating rate (°C/min),
 E_a : activation energy (kJ/mol).

2.6. XRD and FTIR Characterisation

X-ray Diffraction (XRD) analysis was performed using a Bruker D8 Advance diffractometer with Cu K α radiation, scanning from 5 to 80° (2 θ) at a 0.02° step size and a scan rate of 2 °/min to identify the crystalline phases in WSA. Fourier-Transform Infrared (FTIR) spectroscopy was performed using a PerkinElmer Spectrum 100 over 4000-400 cm⁻¹, with 4 cm⁻¹ resolution and 32 scans per sample, to assess the functional groups associated with thermal organic-matter removal and silicate structures. Samples were dried at 105 °C for 24 h, incinerated at 600 °C and ground to pass a 75 μ m sieve before characterisation.

3. Results and Discussion

3.1. Thermal Decomposition Optimisation

Figure 1 shows the thermal stability and decomposition behaviour of WSA in air and nitrogen. Three stages in air were identified: moisture loss at 25-150 °C, primary organic decomposition at 200-500 °C, and higher-temperature carbonate decomposition at 600-900 °C. It has been shown that air produces less residual mass than nitrogen, as the latter has lower oxidation. In a previous study by Mojapelo et al. [11], 600 °C air condition was based on the impact on WSA-concrete. In the present paper, this was considered in the

context of overall atmosphere- and heating rate-dependent thermal data. Figure 2 compares the TGA and DTG responses in air and nitrogen at nominal target temperatures of 320, 400, 500, 600 and 900 °C. These are target temperatures, not heating rates. In air, the three stages of mass loss are the evaporation of moisture below 150 °C, the first organic decomposition between 200 and 500 °C, and mineral decomposition beyond these values. In nitrogen, the DTG is broader and less intense, and the residual mass is higher, which is consistent with the devolatilisation of minerals under oxygen-limited conditions. This comparison extends the thermal profile chosen in the previous study by defining the atmosphere-dependent process response [11].

3.2. Kinetic and Thermodynamic Analysis

The Coats-Redfern kinetic parameters for the thermal decomposition of Polokwane WWS are shown in Table 3. The

activation energies for the fitted air models were 118.6 kJ/mol during stage 2 and 142.1 kJ/mol during stage 3, compared with 94.8 and 107.5 kJ/mol in nitrogen. These values are considered atmosphere-dependent effective parameters in the selected laboratory models.

The Coats-Redfern regression plots in Figure 3 provide the fit statistics used to select the models in Table 4. The reported coefficients of determination range from 0.986 to 0.994. These values indicate good linear fits for the selected models, but do not eliminate uncertainty arising from the single-step model assumption, the use of one primary heating rate, sample heterogeneity, and thermal lag. Accordingly, the parameters are suitable for laboratory-scale comparative analysis and for defining pilot-scale test conditions rather than for claiming industrial-scale process design.

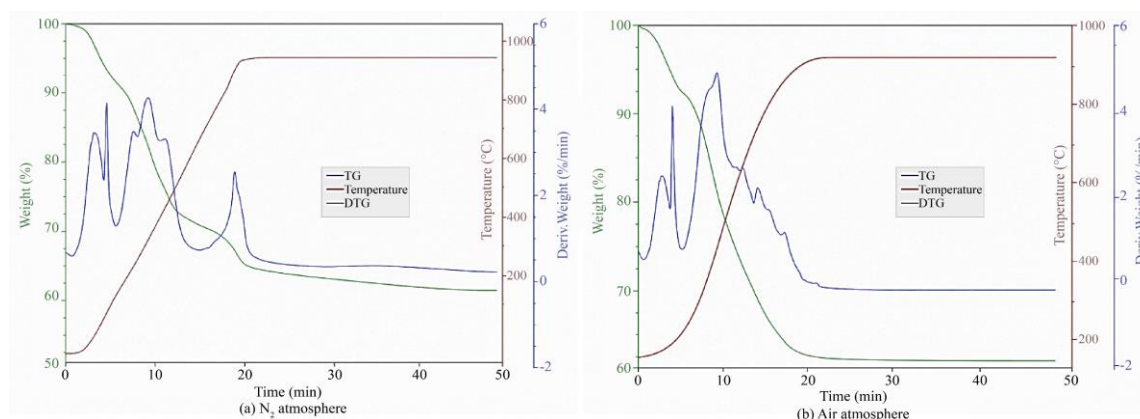


Fig. 1 Thermal stability and decomposition

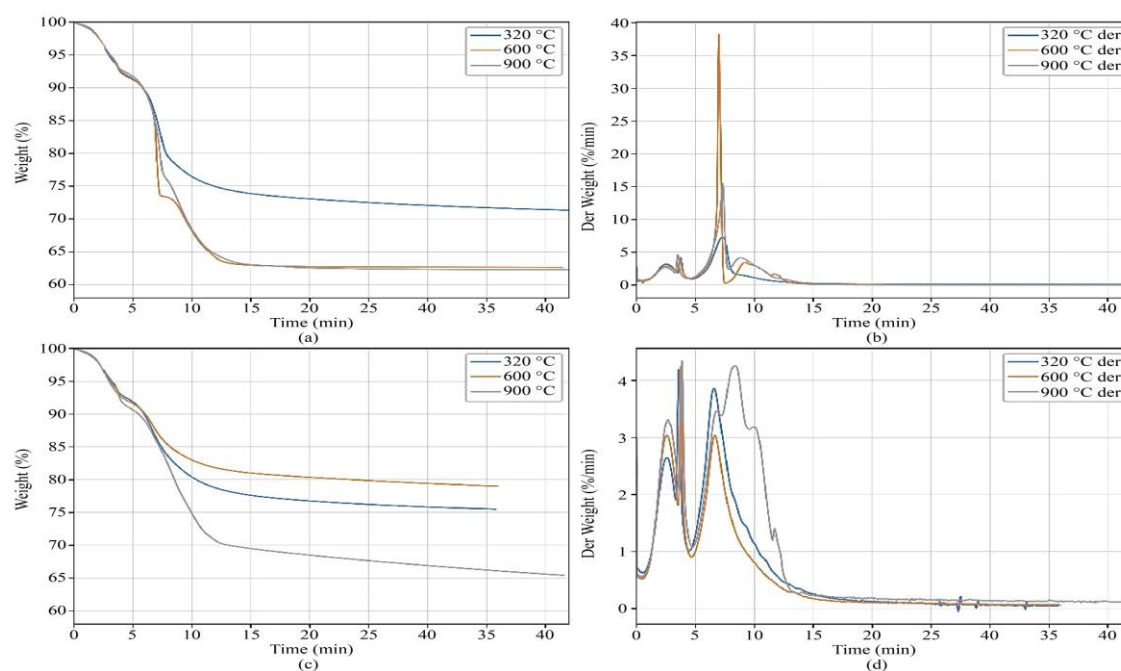


Fig. 2 Thermogravimetric analysis of wastewater sludge under (a) Air and (c) Nitrogen atmosphere, and derivative thermogravimetric analysis of wastewater sludge under (b) Air and (d) Nitrogen atmosphere.

Table 3. Kinetic parameters of Polokwane WWS by the Coats-Redfern method

Stage of Decomposition	Best Fit Model $g(\alpha)$	Activation Energy (E_a) (kJ/mol)	Pre-exponential Factor (A) (min^{-1})	Correlation Coefficient (R^2)
Stage 2 (Air)	(Contracting Volume) $1-(1-\alpha)^{1/3}$	118.6	1.6×10^9	0.994
Stage 3 (Air)	(Avrami-Erofeev A2) $3/2 \cdot (1-\alpha)^{-2/3} - 1$	142.1	2.1×10^{10}	0.991
Stage 2 (N_2)	(Contracting Area) $1-(1-\alpha)^{1/2}$	94.8	3.2×10^7	0.986
Stage 3 (N_2)	(1 st -order F1) $-\ln(1-\alpha)$	107.5	5.9×10^8	0.989

Table 4. Kinetic parameters for WWS thermal decomposition

Decomposition stage	Temperature range ($^{\circ}\text{C}$)	Atmosphere	Energy Activation (kJ/mol)	Kinetic model	R^2	Engineering application
Stage 2: Organic decomposition	200-500	Air	118.6 ± 5.2	R3 (Contracting volume)	0.994	Critical for WSA quality
		N_2	94.8 ± 4.6	R2 (Contracting area)	0.986	
Stage 3: Carbonate decomposition	600-900	Air	142.1 ± 6.8	A2 (Avrami-erofeev)	0.991	Final WSA formation
		N_2	107.5 ± 5.1	F1 (First order)	0.989	

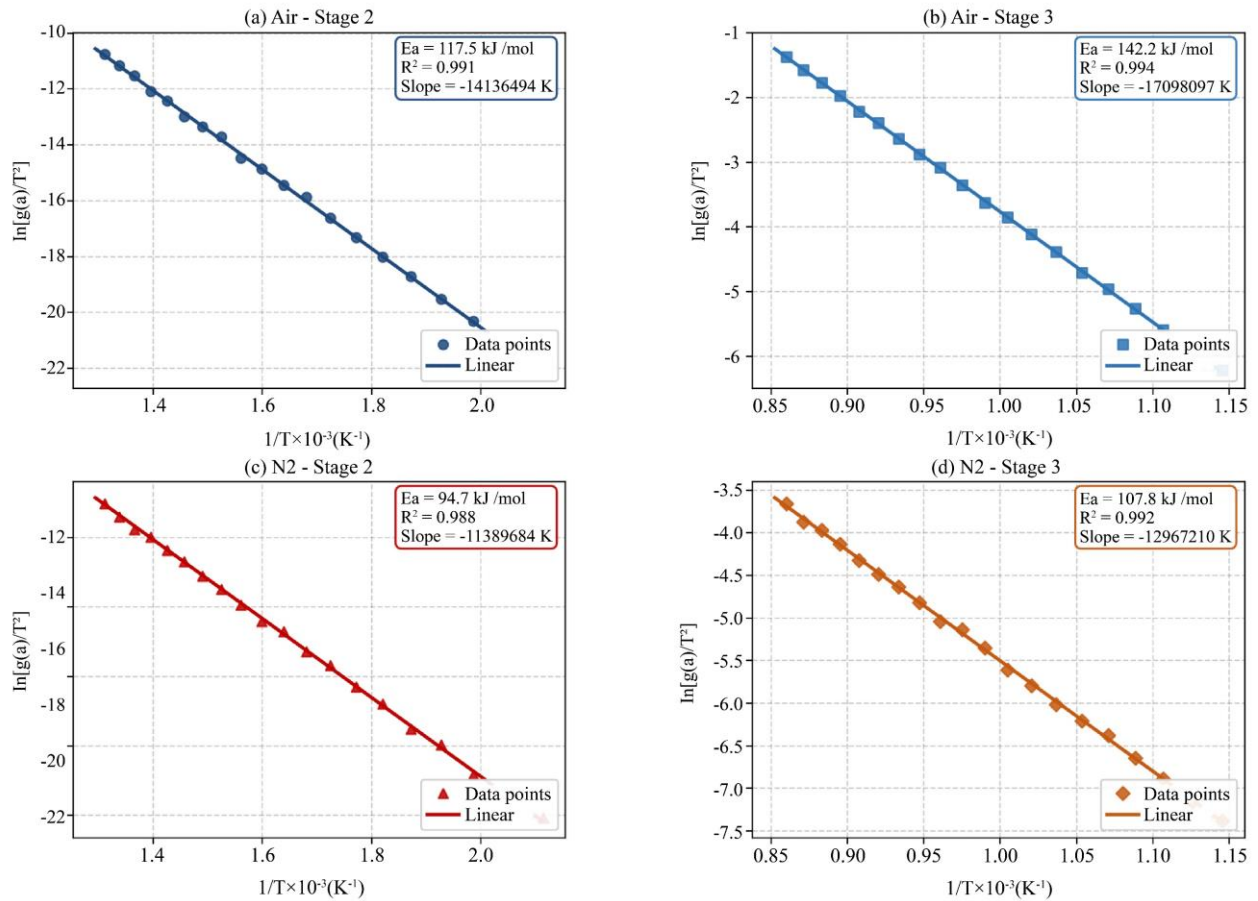


Fig. 3 Coats-Redfern kinetic regression plots for Polokwane WWS thermal decomposition under air and N_2 atmospheres (heating rate: $40^{\circ}\text{C}/\text{min}$). Panels (a)-(d) show $\ln[g(a)/T^2]$ vs. $1/T$ for each decomposition stage.

The fitted activation energies and regression statistics allow comparison of the tested thermal datasets. They can be used to define pilot-scale test parameters, such as heat transfer, gas-solid contact and heating uniformity.

$$t = (1/A) \times \exp(E_a/RT) \times \int g(\alpha) d\alpha \quad (5)$$

The kinetic calculation yields a short, idealised stage time in the given model.

3.3. X-ray Diffraction (XRD)

Following the retained thermal and kinetic evaluation, the XRD patterns of the WWS and of the WSA fired at 500 and 600 °C are shown in Figure 4 to illustrate changes in crystalline phases with increasing thermal treatment temperature. The WWS exhibits weak peaks and broad band

widths, suggesting a significant organic content (~15%) and mostly non-crystalline phase structure [12]. At 500 °C, sharp peaks of quartz (SiO_2), Calcite (CaCO_3), and Hematite (Fe_2O_3) are observed, indicating partial decomposition of organics and a moderate degree of crystallinity (~45%) [16]. The phase-identification discussion is linked to the selected WSA condition used throughout the research program.

At 600 °C, the sharper reflections indicate increased crystalline-phase development. Quartz, calcite and hematite are identified as phase context for the selected ash.

The XRD pattern identifies quartz, calcite and hematite reflections. These retained patterns provide mineral-phase context for the thermal transformation. There is no established pozzolanic reactivity, cement replacement suitability, or concrete performance.

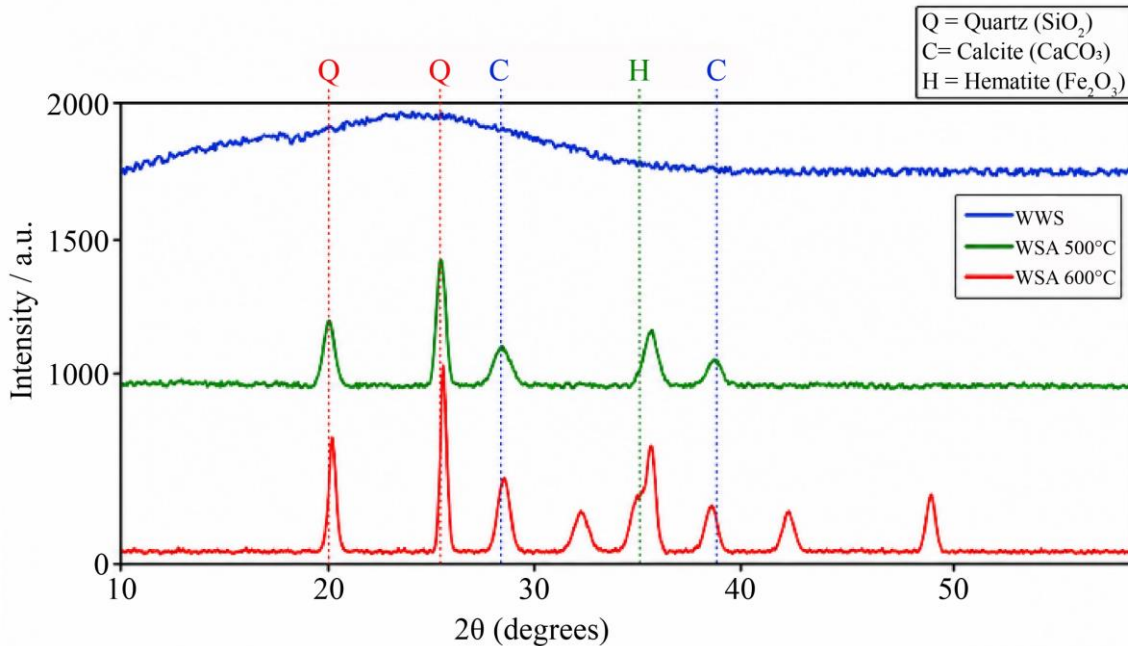


Fig. 4 XRD patterns of WWS and WSA at different temperatures

3.4. Fourier-Transform Infrared Spectroscopy (FTIR)

The FTIR-based quality control method advances the practical monitoring of municipal operators and has closed a void in relatively recent research about waste management [12]. Figure 5 represents the degradation and the preservation of organic matter, as well as mineral mineralisation, with increasing temperature. The C-H stretching bands ($2920\text{--}2850\text{ cm}^{-1}$) became softer at 500 °C and disappeared at 600 °C, indicating that the exposed organics had been thoroughly oxidised.

The temperature-dependent behaviour of organic removal relates well to recent studies optimising the thermal treatment of WWS, which enhanced the strength of FTIR assessments [13]. No C=O/N-H bending bands ($1600\text{--}1400\text{ cm}^{-1}$) were

detected at 600 °C, indicating complete combustion. The increase in the Si-O stretching band (1030 cm^{-1}) occurred as no silicate network decomposition was present, while the O-H band (3400 cm^{-1}) sharpened due to moisture and residual organics escaping.

4. Environmental Considerations

Table 5 depicts the Toxicity Leachability of Polokwane WSA by TCLP (EPA Method 1311), showing good environmental performance for all regulated heavy metals. Concentrations of all eight heavy metals were far below the recommended limits for WSA; listed EPA-regulated metals are compliant within hazardous waste characterisation limits, thereby confirming that the Polokwane WSA does not meet United States Federal criteria for hazardous waste. The

concentrations of Cr, Cu, and Hg were below the analytical method's detection limits, indicating a low leaching potential for these priority pollutants. Importantly, the TCLP results indicate that the levels of zinc at 6.4627 mg/L, compared to the TCLP limit of <7.00 mg/L and nickel at 0.1041 mg/L, compared to the TCLP limit of <7.50 mg/L) are within limits,

addressing two of the most common concerns associated with wastewater materials. The acidic environment (pH = 5.07), characterised by high electrical conductivity (1992.19 $\mu\text{S}/\text{cm}$) and total dissolved solids (1275.00 mg/L), acts as a leaching environment that can enhance the mobility of metals over time.

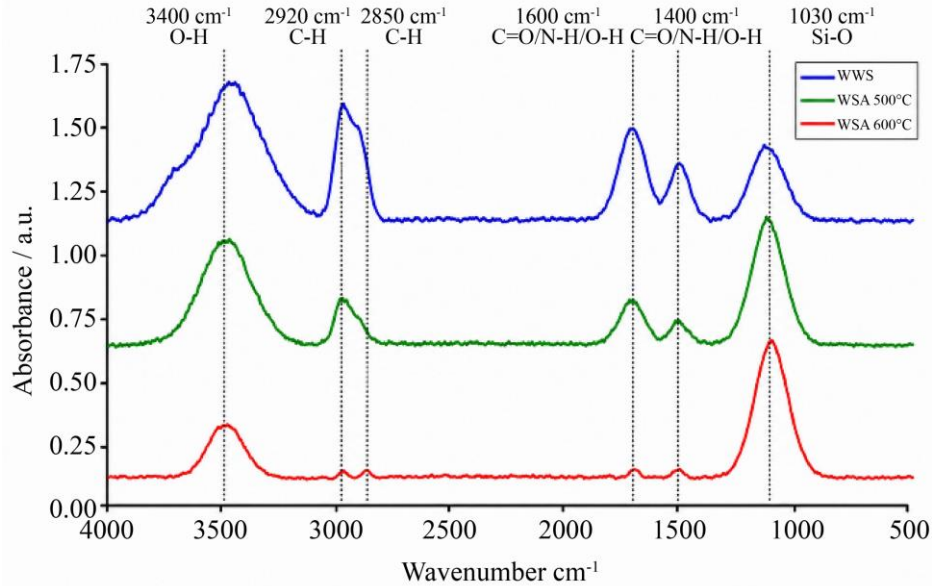


Fig. 5 FTIR spectroscopy to assess the chemical structures of WWS and WSA

Table 5. Leachate characteristics of Polokwane WSA evaluated under TCLP (EPA Method 1311) compared with TCLP acceptable limits and EPA regulatory standards

Parameter	Unit	Polokwane WSA (measured)	TCLP Limit (>10xAE)	EPA TCLP (1992)	Compliance
As	mg/L	0.0447	>3.80	5.00	✓
Cd	mg/L	0.0059	>0.310	1.00	✓
Cr	mg/L	<0.025	>0.200	5.00	✓
Cu	mg/L	<0.010	>1.30	—	✓
Pb	mg/L	0.0029	>1.20	5.00	✓
Hg	mg/L	<0.001	>0.220	0.200	✓
Ni	mg/L	0.1041	>7.50	—	✓
Zn	mg/L	6.4627	>7.00	—	✓
pH		5.0700	—	—	—
EC	$\mu\text{S}/\text{cm}$	1992.19	—	—	—
TDS	mg/L	1275.00	—	—	—

5. Study Limitations and Required Long-Term Validation

The evidence on environmental and durability in this study is limited to WSA TCLP screening and 28-day compressive strength. No monolithic leaching, carbonation, wet-dry cycling, sulphate exposure, chloride ingress, freeze-thaw, or long-term strength-retention tests were performed on WSA concrete. TCLP cannot reproduce the diffusion-controlled leaching and environmental exposure conditions experienced by hardened concrete. Accordingly, the present

results do not demonstrate long-term environmental safety or field durability. Future work should test the 2.5-7.5% WSA mixes using monolithic-leaching and durability protocols under representative South African exposure conditions.

6. Conclusion

The study compares the thermal decomposition of Polokwane WWS under air and nitrogen across the tested heating-rate conditions. Air produced more extensive oxidation and lower residual mass than nitrogen. The Coats-

Redfern analysis gave effective activation energies of 118.6 and 142.1 kJ/mol for Stages 2 and 3 in air, compared with 94.8 and 107.5 kJ/mol under nitrogen. These results provide a laboratory-scale basis for defining pilot-scale thermal tests. The 600 °C air condition and 35 min laboratory protocol require validation at pilot scale and are not presented as an industrial design. XRD and FTIR provide evidence of retained phases and functional groups of thermal transformation. Concrete performance, durability, chemical compliance, and TCLP outcomes from the same research programme are reported in the companion article [1] and are not presented here as independent or newly discovered evidence.

Conflicts of Interest

The authors declare no competing financial interests or personal relationships that could influence the work reported in this study.

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Data Availability Statement

The raw TGA/DTG records, test-condition metadata, and

calculation workflow for the retained thermal numbers and kinetic parameters are available from the corresponding author for editorial review upon reasonable request. The related WSA concrete application data are reported in Mojapelo et al. [11].

Author Contributions

Kobe Samuel Mojapelo: Conceptualisation, formal analysis, Investigation, Methodology, Project administration, Validation, Writing the original draft, Writing review and editing. Everardt Andre Burger: Methodology, Validation, Writing review and editing. Jacques Snyman: Formal analysis, writing review and editing. Idowu David Ibrahim: Conceptualisation, formal analysis, Methodology, Validation, writing of the original draft, writing review, and editing. Charity Maepa: Conceptualisation, formal analysis, Methodology, Validation, writing of the original draft, writing review, and editing.

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