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THERMAL (TGA AND DSC) AND XRD ANALYSIS OF IRON ORE TAILINGS OF KIRIBURU MINES, JHARKHAND

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ABSTRACT

This study investigates the thermal behaviour of Kiriburu iron mine tailings using thermogravimetric analysis (TG), differential scanning calorimetry (DSC), and X-ray diffraction (XRD). The decomposition behaviour was analyzed by tracking weight loss, heat flow variations, and phase transitions under controlled heating conditions. TG results indicate multiple stages of weight loss of approximately 25-26% with distinct decomposition stages, including moisture evaporation, dehydration, Fe-bearing phase breakdown, and high-temperature phase transitions. DSC analysis identifies exothermic peaks at 350 °C and 454.75 °C, along with endothermic peaks at 73.45 °C, 280 °C, and 290.54 °C, indicating phase transformations that reflect the material's thermal stability and its suitability for ceramic applications. XRD confirms the presence of hematite, quartz, and aluminosilicates, which undergo phase transformations at elevated temperatures and amorphous nature, which is effective in controlling the geopolymeric reactions. Overall, the study provides insights into the thermal analysis of iron ore tailings and underscores their potential for high-temperature industrial applications, supporting the sustainable utilization of industrial waste materials.

Keywords: Thermal Analysis, Iron Mine Tailings, TG, DSC, XRD, Phase Transformation.

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INTRODUCTION

Iron ore tailings are a byproduct of ore extraction, which is rich in iron oxides and silica, along with a considerable amount of alumina.[1] To promote sustainable applications, a thorough understanding of their thermal stability and decomposition behavior is essential, particularly for reducing waste accumulation and environmental hazards.[2] Thermal decomposition kinetics is crucial in materials science for predicting the behavior of materials under high-temperature conditions.[3] It involves studying the rate at which materials lose mass and undergo phase transitions, exhibiting thermal reactions when it is subjected to heating, in this way it provides key insights into material stability.[4] Understanding these thermal kinetics decompositions enables the optimization of tailing for applications such as ceramic manufacturing, geopolymer synthesis, and high-temperature refractory materials. Thermal analysis techniques such as TGA and DSC provide crucial information about weight loss patterns, reaction kinetics, and phase transitions.[5] TGA measures the mass loss of a sample as a function of temperature[6], while DSC records heat flow variations, indicating endothermic or exothermic transitions. X-ray diffraction (XRD) further helps in identifying structural transformations at elevated temperatures and monitoring their structural changes upon heating.[7] This study aims to characterize the thermal decomposition kinetics of iron ore tailings to evaluate their thermal stability and suitability for potential engineering applications, particularly in high-temperature material development and industrial processing. Additionally, the study contributes to the Sustainable Development Goals by promoting the reuse of iron ore tailings through the responsible consumption of industrial waste in construction and materials engineering applications.

EXPERIMENTAL

Iron ore tailings were collected from the Kiriburu iron mine tailings dumpsite in the slurry state as per standard guidelines of the Indian standard code for collection of soil samples (IS 2720, 1893). The samples were then dried, ground, and sieved to obtain a uniform particle size for the analysis.

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Thermogravimetric (TG) Analysis

TGA was conducted using a thermogravimetric analyzer (Model: DTG-60) made by Shimadzu Corporation, Japan, at Central Instrumentation Facility, BIT Mesra, Ranchi, under a controlled heating rate. The weight loss of the sample was recorded as a function of temperature, identifying different thermal decomposition stages.

Differential Scanning Calorimetry (DSC)

DSC of iron tailing sample was carried out on the TA Instruments, USA of Model Q 10, for which the temperature ranges between 0.1°C to 500°C per minute. Nitrogen atmosphere was adopted for the analysis to detect exothermic and endothermic reactions. Later, heat flow associated with thermal transitions was analyzed to determine phase transformations.

X-ray diffraction (XRD) Analysis

A high-resolution X-ray diffraction (HR-XRD) on a Rigaku Smartlab diffractometer was used for mineralogical analysis at the central research facility, IIT (ISM) Dhanbad. This instrument utilizes a 3-kilowatt Cu Kα X-ray source, with a scan type of 1D modes, for the scan ranging of 2θ from 5° to 70° with a scan speed of 2° per minute, for a step size of 0.02° was adopted with powder diffraction measurements. The chemical composition was analyzed using Wavelength Dispersive X-ray fluorescence. The analysis was conducted on a Rigaku Primus IV spectrometer. The powdered sample of tailings was prepared as pellets by filling the sample in a container and further pressing the sample for 10 seconds at 10 kN of load.

RESULTS AND DISCUSSION

Thermogravimetric Analysis

The thermogravimetric analysis (TGA) showed a total weight loss of approximately 25-26%, indicating thermal decomposition. The observed weight loss incorporated in Table-1 follows four distinct stages: minimal moisture evaporation (30-50°C), dehydration of iron hydroxides and carbonates (200-400°C), breakdown of Fe-bearing phases (400-800°C), and high-temperature phase transformations (800-1200°C) (Fig.-1). These transformations align with findings from previous studies[8], where similar weight loss trends were observed due to dehydration, oxidation, and silicate restructuring processes (Frontiers in Earth Science, 2023; ISIJ International, 2014).

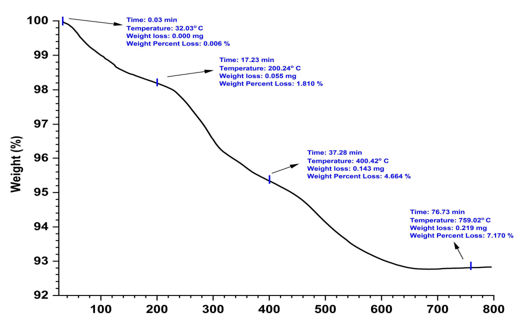

Fig.-1: Weight Loss of the Tailing Sample with Temperature Ranging from Ambient Temperature to 800° C

The data from Table-1 confirms that moisture loss and hydroxide decomposition occur at lower temperatures, while iron phase transitions dominate at higher temperatures, making tailings suitable for refractory applications. Comparatively, literature reports show weight loss rates of 0.28% - 0.36% for moisture loss and dehydration, and 0.32% - 0.36% for Fe-phase decomposition, which are consistent with the present study. Notably, higher weight loss in the current analysis would be due to variations in mineralogical composition and volatile content.

The phase transformations in iron oxides and silicates at 800-1200°C confirm their potential applicability in ceramic production, cement manufacturing, and refractory material synthesis (Fig.-2). These findings show that the utilization of iron mine tailings for high-temperature industrial processes is possible.

Table-1: Summary of Weight Loss Stages in TGA

Stage	Temperature Range (°C)	Weight Loss (%)	Possible Interpretation
Stage 1	30-50	0.006 - 0.135	Moisture evaporation due to physically adsorbed water loss
Stage 2	200-400	4.664	Dehydration of iron hydroxides (goethite → hematite), clay mineral water loss, and carbonate decomposition, releasing CO_2
Stage 3	400-800	4.212 - 7.832	Decomposition of Fe-bearing phases (siderite → hematite) and transformation of iron-silicate minerals
Stage 4	800-1200	7.170 - 12.724	Complete hematite formation, silicate restructuring, and potential sintering effects

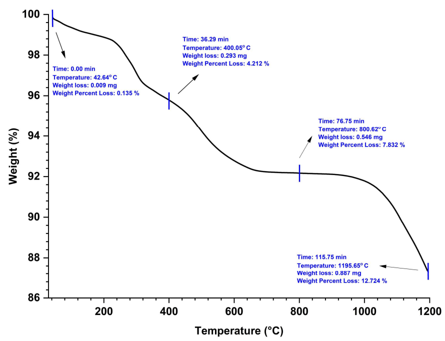

Fig.-2: Weight Loss of the sample beyond 800° C to 1200° C

DSC Analysis

The DSC thermograms showed distinct endothermic and exothermic peaks. The peak temperatures and enthalpy changes indicated phase transitions, including oxidation reactions and structural rearrangements[9] in iron-bearing minerals (Fig.-3). The first endothermic transition occurs at an onset temperature of 51.25°C, reaching a peak at 73.45°C, which is likely associated with moisture loss or an initial phase transition. An endothermic peak at 290.54°C corresponds to the melting or decomposition of organic/inorganic phases. The highest recorded exothermic peak at 454.75°C indicates crystallization or an oxidation-induced structural reorganization. The second endothermic transition occurs at the onset temperature of 58.13°C, having a peak temperature of 69.86°C, followed by another peak temperature of 572.89°C, indicating the transition from wustite to hematite phase transformation (Fig.-4).[10] Figure-5 indicates an onset temperature of 69.29°C with a peak temperature of 80.29°C, and another onset temperature of 287.75°C associated with the highest peak temperature of 585.99°C, suggesting another phase transformation and recrystallization. Further, beyond 1100°C, iron ore tailings first melted to produce the initial liquid phase transitions[11] indicating the ability of a material to mix with other substances to produce materials of industrial importance with good mechanical properties. Liquid phase formation can also promote reduction processes at higher temperatures to produce metallic iron particles. The thermal analysis showed that the material underwent multiple phase transitions, which suggests the presence of thermally unstable components that decompose at moderate to high temperatures. The presence of multiple crystallization peaks indicates that the tailings contain amorphous and crystalline phases, which will influence their engineering applications. The observed high-temperature exothermic peak at 454.75°C implies structural reorganization and oxidation, which is attributed to the presence of iron-rich phases transforming (Fig.-5). Overall, the DSC analysis provides crucial insights into the thermal stability, and the phase transitions suggest that iron tailings could be effectively utilized in thermally stable materials such as geopolymer-based composites and high-temperature ceramics.

XRD Analysis

The diffraction pattern shown in Fig.-6 has multiple peaks corresponding to major mineral phases, which indicates important structural changes upon heating.

The key peaks identified from the XRD spectra are as follows:

- 40.42°, 41.04°, 49.63°, and 54.21° (2-theta) – Corresponding to hematite (Fe_2O_3), confirming the dominance of iron oxide phases.
- 26.80°, 33.33°, 35.79°, and 36.76° – Attributed to quartz (SiO_2), highlighting the presence of silica in the tailings.
- 12.52°, 24.33°, and 25.02° – Associated with aluminosilicates, which contribute to the refractory nature of the material.

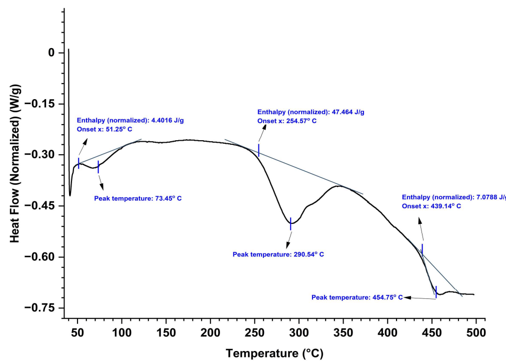

Fig.-3: Heat Flow Variation Confirming Multiple Phase Transformations

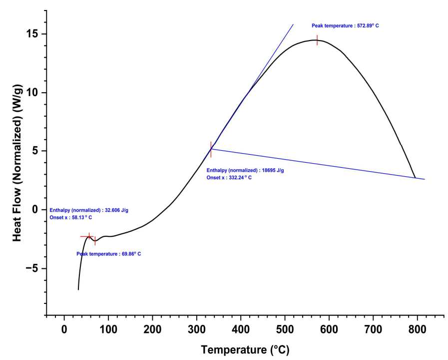

Fig.-4: Heat Flow to the Tailing Sample for Temperature Ranging from Ambient (25° C) to 800° C

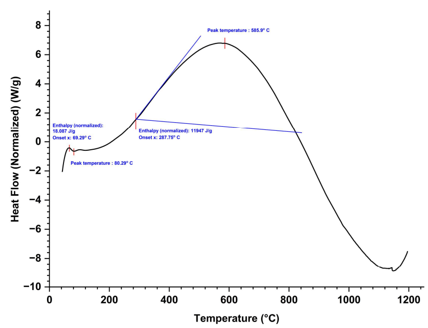

Fig.-5: Heat Flow to the Tailing sample beyond 800° C to 1200° C

These findings confirm the presence of iron-rich phases along with silicates and aluminosilicates that undergo phase transitions under thermal conditions.[10] The presence of hematite and quartz suggests potential in high-temperature construction materials, as these minerals contribute to durability[11] and mechanical strength under extreme conditions. A highly amorphous nature is shown by the XRD pattern, which is effective in controlling the geopolymeric reactions.

CONCLUSION

This study provides a comprehensive analysis of the thermal decomposition behavior of Kiriburu iron mine tailings, utilizing TGA, DSC, and XRD techniques. The key findings include. A total weight loss of 25-

26%, with distinct thermal decomposition stages observed in the TGA results, highlighting moisture loss; carbonate decomposition, and Fe-bearing phase transformations.

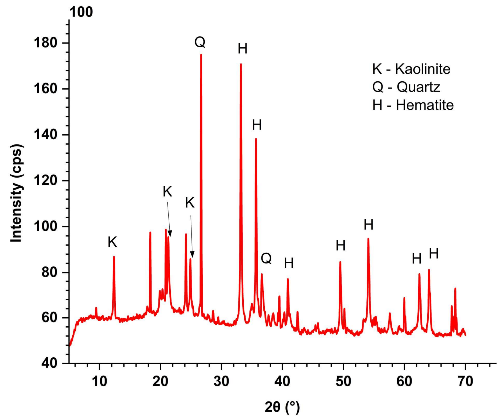

Fig.-6: XRD Peaks of Iron Tailings Sample

DSC analysis confirmed multiple endothermic and exothermic reactions, indicating structural reorganization, crystallization, oxidation of iron-bearing phases, and Liquid phase transformation, indicating the ability to produce industrial materials. XRD results identified hematite, quartz, and aluminosilicate as primary mineral phases, confirming high-temperature structural transformations and the amorphous nature of materials. These findings indicate that iron ore tailings exhibit promising thermal stability and phase change characteristics, making them suitable for high-temperature applications in industries such as ceramic manufacturing, geopolymer synthesis, and refractory material production. The study highlights the potential for waste utilization in engineering applications, contributing to sustainable construction practices.

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CONFLICT OF INTERESTS

The authors declare that there is no conflict of interest.

AUTHOR CONTRIBUTIONS

The present work is related to *SDG-12: Responsible Consumption and Production*. All the authors contributed significantly to this manuscript, participated in reviewing/editing and approved the final draft for publication. The research profile of the authors can be verified from their ORCID ids, given below:

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REFERENCES

1. C. Wang, J. Jing, Y. Qi, Y. Zhou, K. Zhang, Y. Zheng, Y. Zhai, and F. Liu, *Frontiers in Earth Science,* **11**, 1181984(2023), https://doi.org/10.3389/feart.2023.1181984

2. M. Edraki, T. Baumgartl, E. Manlapig, D. Bradshaw, D. M. Franks, and C. J. Moran, *Journal of Cleaner Production,* **84**, 411(2014), https://doi.org/10.1016/j.jclepro.2014.04.079
3. S. Vyazovkin, A. K. Burnham, J. M. Criado, L. A. Pérez-Maqueda, C. Popescu, and N. Sbirrazzuoli, *Thermochimica Acta,* **520**, 1(2011), https://doi.org/10.1016/j.tca.2011.03.034
4. T. Ozawa, *Journal of Thermal Analysis,* **2**, 301(1970), https://doi.org/10.1007/BF01911411
5. H. E. Kissinger, *Analytical Chemistry,* **29**, 1702(1957), https://doi.org/10.1021/ac60131a045
6. J. H. Flynn and L. A. Wall, *Journal of Polymer Science Part B: Polymer Letters,* **4**, 323(1966), https://doi.org/10.1002/pol.1966.110040504
7. B. D. Cullity and R. Smoluchowski, *Physics Today,* **10**, 50(1957), https://doi.org/10.1063/1.3060306
8. Y. Qu, Y. Yang, Z. Zou, C. Zeilstra, K. Meijer, and R. Boom, *ISIJ International,* **54**, 2196(2014), https://doi.org/10.2355/isijinternational.54.2196
9. S. Vyazovkin, V. Rives, and C. Schick, *Thermochimica Acta,* **500**, 1(2010), https://doi.org/10.1016/j.tca.2010.02.005
10. M. J. Anju, M. Beulah, and A. Varghese, *International Journal of Pavement Research and Technology,* **1**, 20 (2024), https://doi.org/10.1007/s42947-024-00446-8
11. R. M. Cornell and U. Schwertmann, *Corrosion Reviews,* **15**, 533(1997), https://doi.org/10.1515/CORRREV.1997.15.3-4.533

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