STUDIA ECOLOGIAE ET BIOETHICAE
2024, vol. 22, no. 4:
p-ISSN 1733-1218; e-ISSN 2719-826X
DOI: http://doi.org/10.21697/seb.5826

Characterising Rice Straw Ash: Unlocking the Potential of Agricultural Residues

Właściwości popiołu ze słomy ryżowej: wykorzystanie potencjału odpadów rolniczych

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Received: 24 Jul 2024; Revised: 04 Oct, 2024; Accepted: 08 Oct, 2024;
Pre-published 10 Nov, 2024

Abstract: Air pollution has become a scourge to contend with in India. The recorded concentrations of particulate matter (PM) in the atmosphere, the unabated emission of pollutants from vehicular exhausts, and recurring episodes of extremely poor condition (AQI>300) in the winter months, have rightfully and necessarily, spurred efforts in the industrial, governmental and research spheres to alleviate its detrimental impacts. Various point sources like biomass burning, coal combustion for power generation, and traditional agricultural practices such as stubble burning, collectively contribute to a steady rise in ambient particulate matter (PM) pollution. This study focuses on the utilization of rice straw – an abundant agricultural residue in a country like India – motivated by promoting and contributing to the soil-to-soil circularity paradigm. It encompasses the characterisation of straw ash from the rice, by delineating its physical properties, thermal characteristics, and chemical composition with the help of Thermogravimetric analysis (TGA), X-ray diffraction (XRD), Electron Probe Microanalysis (EPMA), Scanning Electron Microscopy (SEM), Transmission Electron Microscopy (TEM) and Dynamic Light Scattering (DLS). The results indicate that rice straw ash (RSA) possesses high silica content and favorable thermal stability. The RSA exhibited a porous structure, which enhances nutrient adsorption and microbial activity. Its incorporation into soil significantly improved soil nutrition and health, promoting a more sustainable agricultural practice. Entrenching this soil-to-soil thinking will contribute directly and indirectly to a host of sustainable development goals in a future Indian circular bioeconomy.

Keywords: rice straw ash, soil fertility, stubble burning, valorisation, circular economy, biomass burning

Streszczenie: Zanieczyszczenie powietrza jest obecnie poważnym problemem w Indiach. Rejestrowane duże stężenia pyłu zawieszonego (PM) w atmosferze, ciągła emisja zanieczyszczeń ze spalin samochodowych i powtarzające się epizody wyjątkowo złego stanu powietrza (AQI>300) w miesiącach zimowych słusznie i koniecznie zainicjowały działania w sferze przemysłowej, rządowej i badawczej w celu złagodzenia ich szkodliwego wpływu. Różne źródła emisji zanieczyszczeń, takie jak spalanie biomasy, spalanie węgla w celu wytwarzania energii i tradycyjne praktyki rolnicze, takie jak spalanie ściernisk, łącznie przyczyniają się do stałego wzrostu zanieczyszczenia powietrza pyłem zawieszonym (PM). Niniejsze badanie koncentruje się na wykorzystaniu w obiegu zamkniętym (cyrkularność gleba-gleba) słomy ryżowej, która w dużych ilościach pozostaje na polach uprawnych w kraju takim jak Indie. W pracy przedstawiono charakterystykę popiołu ze słomy ryżowej poprzez określenie jego właściwości fizycznych, termicznych i składu chemicznego za pomocą analizy termograwimetrycznej (TGA), dyfrakcji rentgenowskiej (XRD), mikroanalizy sondy elektronowej (EPMA), skaningowej mikroskopii elektronowej (SEM), transmisyjnej mikroskopii elektronowej (TEM) i dynamicznego rozpraszania światła (DLS). Wyniki wskazują, że popiół ze słomy ryżowej (RSA) charakteryzuje się wysoką zawartością krzemionki i korzystną stabilnością termiczną. RSA posiada porowatą strukturę, która zwiększa adsorpcję składników odżywczych i aktywność mikrobiologiczną. Wprowadzenie popiołu do gleby znacznie poprawiło jej żyzność, tym samym tę praktykę można zaliczyć do bardziej zrównoważonych. Promowanie tej praktyki przyczyni się do przyspieszenia rozwoju indyjskiej biogospodarki o obiegu zamkniętym oraz bezpośrednio i pośrednio do osiągnięcia wielu celów zrównoważonego rozwoju.

Słowa kluczowe: popiół ze słomy ryżowej, żyzność gleby, spalanie ściernisk, waloryzacja, gospodarka o obiegu zamkniętym, spalanie biomasy

Introduction

This paper focuses on the valorisation of agricultural residues, particularly rice straw, in the context of India's agricultural sector. It explores the potential of utilizing these residues to produce valuable products such as bioenergy and biochemicals through a circular bioeconomy approach. The motivation behind this research lies in addressing several Sustainable Development Goals (SDGs), including SDG 9 (Industry, Innovation, and Infrastructure), SDG 3 (Good Health and Well-being), and SDG 13 (Climate Action) (Kapoor et al. 2020). In Figure 1, the enabling SDGs (e.g., SDG 9, SDG 17) are depicted at the right, indicating their role in facilitating or supporting the achievement of other SDGs. These enabling SDGs serve as foundational pillars that contribute to the progress of the enabled SDGs (e.g., SDG 2, SDG 3, SDG 7, SDG 12, SDG 15), which are positioned on the left. By valorising agricultural residues through innovative processes, this research contributes to the advancement of sustainable industries, promoting the development of new technologies and practices for converting waste into valuable resources (Chilakamarry et al., 2022). This, in turn, helps mitigate air pollution and associated health risks, particularly in rural areas where agricultural activities are prevalent. Additionally, by reducing reliance on fossil fuels and minimizing waste generation, the utilization of agricultural residues for bioenergy production contributes to climate change mitigation efforts, aligning with the goals of achieving climate resilience and sustainability in agricultural systems.

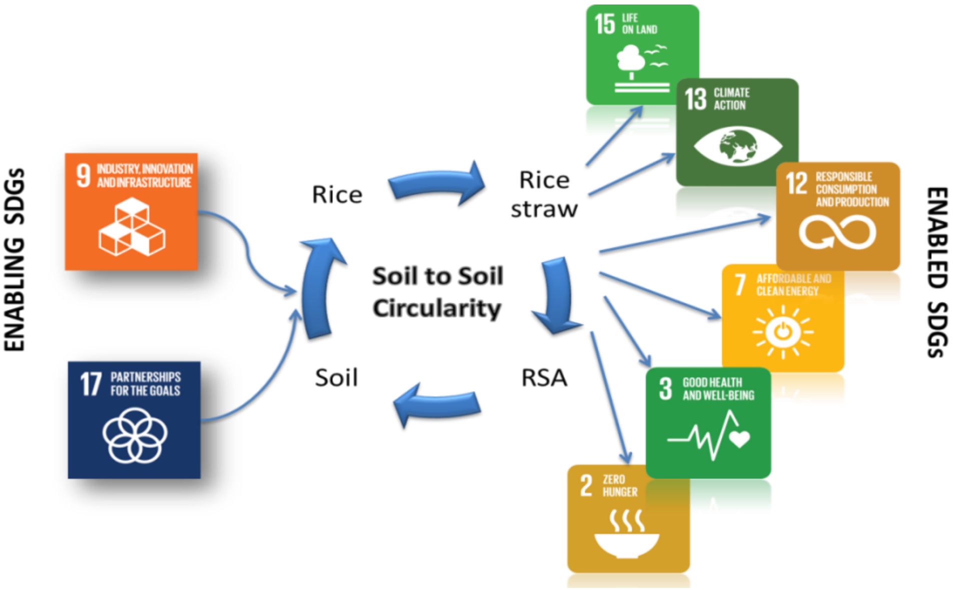


Figure 1: Illustrating the interdependence between enabling SDGs and enabled SDGs.

The circularization of agricultural waste streams not only enhances resource efficiency but also supports economic development by creating new opportunities for rural communities engaged in agriculture (Duque-Acevedo et al. 2020). Through collaborative efforts involving stakeholders from the government, research institutions, and the private sector, this research aims to catalyze the transition towards a more sustainable and circular bioeconomy, fostering environmental management and social equity while driving economic growth. The study focuses on agricultural regions in India, where rice cultivation is prevalent and generates significant amounts of rice straw as waste (Brandão and Santos 2024). These regions serve as potential sites for implementing circular bioeconomy strategies to valorize agricultural residues through a multi-disciplinary approach, combining knowledge from agriculture, biochemistry, and environmental science (Toplicean and Datcu 2024). Materials and methods involve the characterization of agricultural residues, optimization of conversion processes, and economic analysis to assess the feasibility and sustainability of circular bioeconomy practices. Stakeholders involved include farmers, policymakers, researchers, and industries in the agriculture, energy, and environmental sectors. Collaboration among these stakeholders is essential for the successful implementation of circular bioeconomy initiatives in the Indian agricultural sector. In conclusion, this research presents a comprehensive exploration of the potential of agricultural residues to contribute to a sustainable and circular bioeconomy (Dwivedi et al. 2019).

India has been, is, and will continue to have a sizable agro-based economy with regular cultivation of various crops, and in the process, generation of large quantities of organic residues. India is the second-largest wheat producer in the world, and it can ensure food and nutrition security for its vast population (in its pursuit of Sustainable Development Goal # 2) despite its diverse agroecological conditions. In India, wheat cultivation is done during the *Rabi* season – sown from November and harvested between March and April. Similar to wheat, rice paddy cultivation is also an essential component of the primary sector of the Indian economy, and rice is a part of the staple diet of Indians around the country. In the absence of adequate sustainable management practices, large quantities of crop waste are burned openly on the fields every year, occasioning particulate matter (PM) emissions and consequent air pollution (Bhuvaneshwari et al. 2019). The traditional practice of open-field stubble burning contributes significantly to air pollution, releasing harmful particulate matter (PM2.5 and PM10), carbon dioxide, methane, and other toxic pollutants, worsening India's already critical air quality (Bhattacharyya et al. 2021; Farid et al. 2022). The variety of crops cultivated is very wide and includes rice, wheat, sugarcane, cotton, jute, coarse cereals like *sorghum* (broomcorn), maize, millet and barley, oil-yielding crops like soybean, sunflower, rapeseed, mustard, groundnut and castor seed, and pulses like *moong* (green gram), *urad* (black gram), pigeon-pea etc. (Purohit & Dhar, 2015, pp.14–19). What is intuitively obvious but has never actually stood out in importance is the fact that on average 500 metric tons of crop residue generated annually in India (rice and wheat accounting for significant slices of the pie), exceeds the mass of harvested crop available for sale in the marketplace. Abdurrahman et al. (2020) have pointed out that the mass of rice and wheat residues, in general, is 50% greater than that of the wheat and rice grains produced. Stubble burning, while adversely impacting the atmosphere, also harms the pedosphere. In other words, the fertility of the soil is gradually but surely depleted over time. Stubble burning is thus a hotspot process to be addressed with urgency, to truncate the social and environmental footprints of rice and wheat cultivation, particularly, and agriculture in general.

Crop residue production worldwide varies significantly depending on factors such as agricultural practices, crop types, and regional climatic conditions. Globally, billions of tons of crop residues are generated annually, primarily consisting of stalks, leaves, husks, and other post-harvest leftovers. According to Verardi et al. (2023), major crop residues include those from cereals like wheat, rice, maize, and barley, and oilseed crops like soybean and rapeseed. While global annual wheat and rice straw residue production exceeds 700 million metric tons each, maize chips in with more than 1 billion metric tons. This is, quite obviously, a global phenomenon, with the lens trained particularly on the USA, Spain, India, China, and Brazil figuring among the top-five in the world. It has long been known that these crop residues represent a vast and underutilized resource that can potentially be converted into valuable products through novel valorisation-based approaches, contributing to sustainable agriculture and bio-based industries in a much-desired circular bioeconomy in the future. Application of this knowledge has now become imperative.

Basmati (derived from Hindi; and meaning ‘fragrant’) stands out as a distinctive speciality rice variety because of its specific kernel dimensions which contribute to improving its texture when cooked and extending its shelf life in godown and home. According to research conducted by Patlavath and Albert (2021), in the year 2018-19, India exported more than four million metric tons of *Basmati* rice (worth about 4.7 billion USD) to the USA, Saudi Arabia, and several Gulf and European countries. India, at the time of writing, is the largest cultivator and exporter of Basmati rice (Singh et al. 2018). In India, twelve rice varieties (or sub-varieties of *Basmati* rice, to be more specific) are recognized as 'true *Basmati* rice' under the Seed Act 1966; thereby being prioritized for export. PB-1121 (also called the *Pusa Basmati*) and PB-1509 (two of the 12) are cultivated in the northern Indian states of Delhi, Haryana, Himachal Pradesh, Punjab, Uttar Pradesh, and Uttarakhand. Punjab and Haryana emerged as the primary producers of the aforenamed varieties of *Basmati* rice in 2019, as per Patlavath and Albert (2021), and the Agricultural & Processed Food Products Export Development Authority (2023). High-yielding PB 1121 has a length of growing period (LGP) in the range of 140–145 days, vis-à-vis 160 days for the traditional *Basmati* rice, as per Singh et al. (2018). As a result, resource usage - fertilisers and pesticides, water for irrigation -per unit output, is marginally less. Apart from economising cultivation, PB 1121 also provides farmers with slightly more turnaround time to prepare for the timely sowing of wheat, in the alternating rice-wheat cropping system.

While the earlier paragraphs focused on the ‘sources’ of crop residues, the following ones present a synopsis of research conducted on the valorisation of the said residues, and the applications the valorised bio-products can be put to, in a robust circular bioeconomy in the future. A recent study – Zaid et al. (2021) - shows that since rice husk ash is pozzolanic in nature, it serves as a good input in cement production. Characterisation of the rice straws of different varieties of the crop – biochemical, chemical, and morphological - has been of interest to many researchers in the recent past. Bhattacharyya et al. (2020) and Rosado et al. (2022) have reported the presence of cellulose, hemicellulose, and lignin in rice straws. Rice straw ash (RSA) contains potassium oxide and silicon dioxide (or silica), inter alia. Silicon is abundant in agricultural soils and is known to be a biological stimulant which positively influences the growth of plants in general (and some plants in particular). As the agricultural output keeps increasing, the arable pedosphere which is a fund resource (which implies that it is not unconditionally renewable) suffers a depletion of silicon. A tipping point is reached as the ‘carrying capacity of the soil with respect to its silicon content’ witnesses a downward trend. This has an undesirable impact on plant growth (Jin et al. 2020; Savvas and Ntatsi 2015). Valorising rice straw (RS) into either rice straw biochar (RSB) or rice straw ash (RSA) and utilizing these bio-products in agriculture is an eco-friendly and sustainable approach to enhance both soil fertility and crop yield, while obviating a host of other health-related and environmental concerns which were alluded to earlier in this section.

For sustainable agricultural practices, the management of agricultural waste within a circular bio-based economy emerges as a pivotal challenge with profound implications. The major challenge is resource optimization and technological innovation, where maximizing the utilization of agricultural waste while minimizing environmental impact at the same time, stands as a paramount two-fold objective. However, a circular bio-based economy is riddled with obstacles that transcend technologies. Policy and regulatory frameworks in the country (a vital fourth dimension of sustainable development) play a crucial role in shaping the trajectory of sustainable waste management practices.

According to G Venkatesh (2021) biorefineries are the paradigms of the future, capable of utilising a variety of inputs (organic residues) and producing a range of different valorized outputs intended for different applications, by availing of multiple technologies (or suitable combinations thereof). For instance, Ginni et al. (2021) examines biochemical, thermochemical, and hybrid processes in a biorefinery, each with its distinct advantages and challenges. Enzymatic hydrolysis, for example, can convert up to 80% of agricultural residues into sugars, which can then be further processed into biofuels, biochemicals, and biopolymers. Fermentation processes can achieve conversion efficiencies of over 90% in some cases. Devi et al. (2022) have contended that pyrolysis and gasification can entrench themselves in the future, and yield bioenergy and biochar, of agricultural-residue provenance. Pyrolysis can convert up to 70% of biomass into bio-oil (liquid fuel), biochar (solid fuel or soil amendment), and syngas (gaseous fuel), depending on the operating conditions. While these processes offer high conversion efficiencies, they also require careful management to minimize the formation of undesirable environment-damaging and/or health-impacting byproducts.

A significant aspect highlighted in a handful of recently conducted and published studies like Yaashikaa et al. (2022) for instance, is the considerable percentage of agricultural waste (of the more than 500 million tons generated annually) available for biogas production. Research conducted by Kapoor et al. (2020) explores various biogas production technologies, including anaerobic digestion, which is identified as a potential method for converting agricultural waste into biogas where anaerobic digestion has been found to achieve high biogas yields, with methane content ranging from 50% to 70%, depending on the feedstock and process conditions. Additionally, the study highlights the potential for decentralized biogas plants, which can be established in rural areas near agricultural sources, minimizing transportation costs and promoting local energy production. There has been a growing interest in incorporating RSA into industrial applications, such as in concrete production, to improve material strength and sustainability (Bassi et al. 2023). Government-led initiatives, like the promotion of the Pusa Decomposer, aim to accelerate the decomposition of rice straw into nutrient-rich compost, further mitigating the need for open burning (Bhatnagar 2020). The ongoing research on RSA emphasizes its potential for reducing the environmental footprint of agriculture while contributing to a circular bioeconomy, thus aligning with national and global sustainability goals.

The utilisation of RSA as a soil additive – which is the crux and theme of this particular article and the research on which it is based - has not been well researched in the past. It can be reiterated time and again that soil is a fund resource which will be degraded eventually unless the way humans utilise it to feed themselves is changed drastically. Hence, the objective of this research is to investigate the valorisation of rice straw (RS) into rice straw ash (RSA) that can be transformed from a byproduct into a valuable resource, aligning with principles of sustainability, the sustainable development goals (SDGs) and circular economy practices.

1. Methods and materials

1.1. Sample preparation

In this study, rice straw of *Pusa Basmati* (PB 1121) was collected from the Indian Agricultural Research Institute (IARI) in New Delhi, and stored in a controlled laboratory environment. IARI plays a stellar role in crop standardization in the country, through its research in various aspects of agriculture, including crop improvement, agronomy, and soil science. The said rice straw samples were stored at 4°C in sealed plastic bags until they were subjected to the tests. After being allowed to sun-dry for 2 days, to evaporate the moisture in them, thermogravimetric analysis (TGA) was carried out using a PerkinElmer Thermal Analyzer. The sample-mass was measured accurately using the sensor connected to the sample carrier, and recorded as 3.617 mg. It was then placed in an alumina crucible, and nitrogen gas was subsequently purged at a volumetric rate of 100 ml per minute inside the thermal analyzer to evacuate it (Sakhiya et al. 2021). Finally, the sample was heated at a rate of 10°C/min, from room temperature up to

800°C. Nitrogen supply was maintained throughout the reaction to maintain an oxygen-free environment inside the reaction chamber.

Figure 2: From left to right, muffle furnace set at 700 °C, the dried rice straw (RS) sample and the rice straw ash (RSA).

To obtain the RSA, combustion of the Rice Straw (RS) was performed in the muffle furnace at a decided temperature according to the TGA results (refer Figure 3). It was observed from the TGA curve that rice straw was completely degraded at 700°C. According to published literature, the maximum temperature of the furnace can reach anywhere between 650°C to 700°C, to yield carbonized rice straw (Guzmán et al. 2015). The heating rate was kept constant until the temperature reached 700°C (Figure 2), and the temperature was maintained for a residence time of at least 3 hours to ensure the total decomposition of the organic matter. Thereafter, the ash was carefully collected with the help of a spatula. For the characterisation of RSA, SEM and TEM were employed to observe the morphology, topography, and structure of the particles. Dynamic Light Scattering (DLS) was used to determine the particulate sizes. Additionally, XRD analysis was conducted to identify the mineralogical phases present in the ash, with peaks corresponding to quartz, and other crystalline structures. EPMA was also used to determine the elemental composition. Figure 3 illustrates the characterisation exercises conducted on the RSA in this study, and the results of the analyses are presented and discussed in the sub-sections that follow.

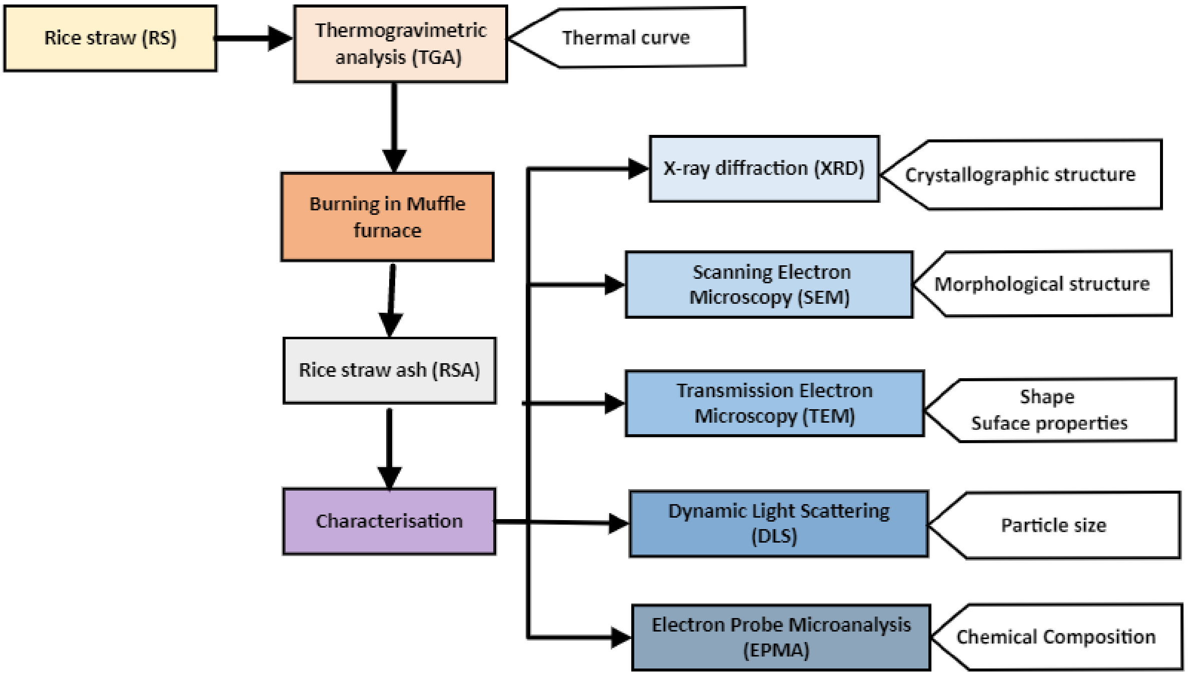


Figure 3: The flow diagram of characterisation and analytical techniques performed via TGA, XRD, DLS, EPMA, SEM and TEM to examine the properties of RS and RSA.

2. Results and Discussion

2.1. Thermogravimetric analysis (TGA)

The TGA curve (refer Figure 4) depicts the gradual reduction in the sample's mass as the temperature rises from 50°C to 800°C at a heating rate of 10°C per minute. Different phases and observations from the TGA curve are as follows:

Initial Phase (50°C to 100°C): In the initial phase, the temperature rises from 50°C to around 100°C. During this period, a noticeable decrease in weight is observed. This weight loss, approximately 7.509%, is attributed to the evaporation of moisture content. Rice straw is hygroscopic in nature, meaning it readily absorbs moisture from the environment. The observed weight reduction is primarily due to the drying process, where both surface-bound and loosely bound water molecules are evaporated (Said et al., 2013. This initial phase indicates the removal of moisture, which is crucial for preparing the sample for subsequent decomposition phases.

Devolatilization Phase (100°C to 300°C): As the temperature increases beyond 100°C and approaches 300°C, the TGA curve shows a significant weight loss. This phase is associated with the devolatilization of rice straw. The decomposition of hemicellulose and cellulose, which are the primary components of the rice straw, occurs in this temperature range (Guzmán et al., 2015). Hemicellulose decomposes first, followed by cellulose. This decomposition process releases volatile compounds (VCs) into the atmosphere. The substantial weight loss observed in this phase is due to the breakdown of these biopolymers. The derivative weight curve, which shows the rate of weight loss, indicates a peak within this range, highlighting the most active phase of decomposition.

Lignin Decomposition Phase (300°C to 460°C): Beyond 300°C, the TGA curve continues to show weight loss, albeit at a slower rate compared to the previous phase. This phase corresponds to the decomposition of lignin, a complex and thermally stable polymer found in rice straw. Lignin decomposes over a broader temperature range, contributing to a more gradual reduction in mass. The weight loss in this phase is due to the release of additional volatile compounds from lignin. The derivative weight curve shows a smaller peak in this range, indicating the slower and more extended decomposition process of lignin compared to cellulose and hemicellulose.

Final Phase (460°C to 800°C): As reported by Sakhiya and fellow researchers (2021) after 460°C, the rate of mass loss decreases significantly, and the TGA curve approaches a plateau. The weight loss becomes almost negligible around 600°C, indicating the completion of major thermal decomposition reactions. At this point, the remaining mass primarily consists of inorganic minerals such as silica, which are thermally stable at high temperatures. The final residual weight, as indicated by the TGA curve, represents the ash content of the rice straw sample.

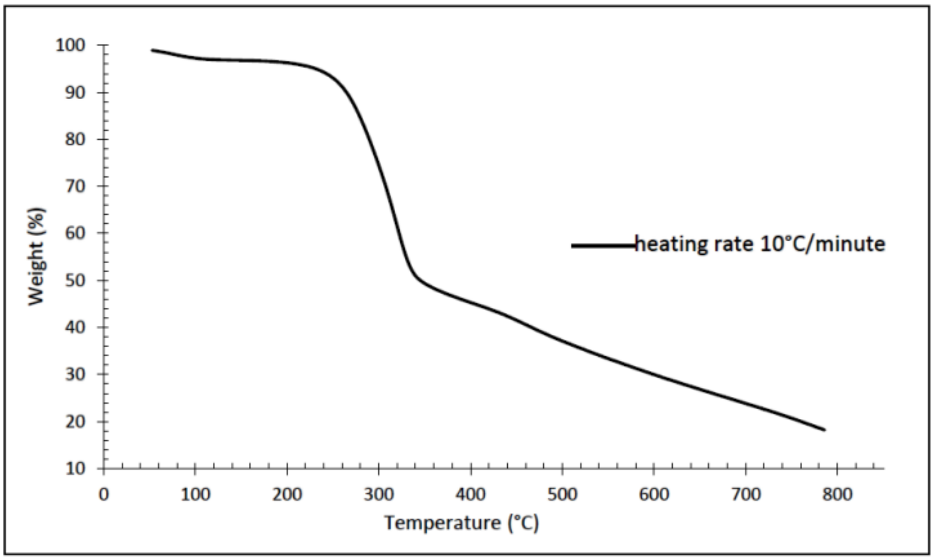


Figure 4: TGA curve showing weight loss (in %) of Rice Straw over time at a constant heating rate from 50°C to 800°C at 10°C per min.

In this study, RS and RSA were characterised from microscopic, chemical composition and reactivity perspectives. The reactivity outcomes of RS help in understanding the thermal behaviour of rice straw, which is vital information from the point of view of bioenergy production. It aids in the identification of key parameters such as ignition temperature, char formation, and combustion kinetics. This knowledge enables the design of highly efficient, low-emitting combustion systems (pyrolysis and gasification). Analysing weight loss patterns and temperature profiles enables the prediction of yields for biofuels, biochar, and other valuable bio-products derived from RS. Additionally, TGA coupled with other analytical techniques like XRD (refer the next sub-section), provides insights into the composition and properties of RSA. Since the intention is to add RSA to the soil and improve its health, information obtained from TGA is useful for the assessment of ash fouling and slagging tendencies in combustion systems.

2.2. X-ray diffraction (XRD) analysis

XRD was performed to analyse RSA qualitatively, and study the mineralogical phases present in the sample (Refer Figure 5). The analysis was conducted from a start angle of 5° to an end angle of 85°, with a step angle of 0.02° and a dwell time of 1.19998 seconds per step, resulting in a total of 4001 data points. The maximum recorded intensity was 302 counts, with a cumulative total count of 178,068 counts. The XRD pattern reveals several sharp peaks superimposed on a broad background, indicative of the sample's mixed-phase nature. The most prominent peak, with an intensity of 100%, is located at approximately 20° 2θ. Other significant peaks are observed around 25°, 35°, and 43° 2θ. The variation in peak intensities suggests differing amounts of crystalline phases within the sample. The highest peak intensity recorded is 302 counts, and the overall pattern displays a mixture of sharp peaks and a broad background, signifying the presence of both crystalline and amorphous phases.

Prominent peaks at approximately 20°, 25°, 35°, and 43° 2θ suggest the presence of quartz (a form of silica), calcite (calcium carbonate), and potentially phases of alumina or hematite, the peak at around 20° 2θ likely corresponds to quartz, while the peak at 25° 2θ matches with the (101) plane of quartz (Pandey et al. 2021) and the peaks at 35° 2θ and 43° 2θ could correspond to alumina or hematite phases. This identification can be achieved by matching the observed peaks with standard reference patterns from databases such as the International Centre for Diffraction Data (ICDD).

Analyzing the intensity and area of the diffraction peaks allows for quantitative phase analysis. This method enables the determination of the relative amounts of different crystalline phases within the sample. A high intensity of sharp peaks indicates a higher degree of crystallinity, whereas a pronounced broad background is indicative of a substantial amorphous phase. The presence of both crystalline and amorphous phases can significantly impact the material's performance in various applications.

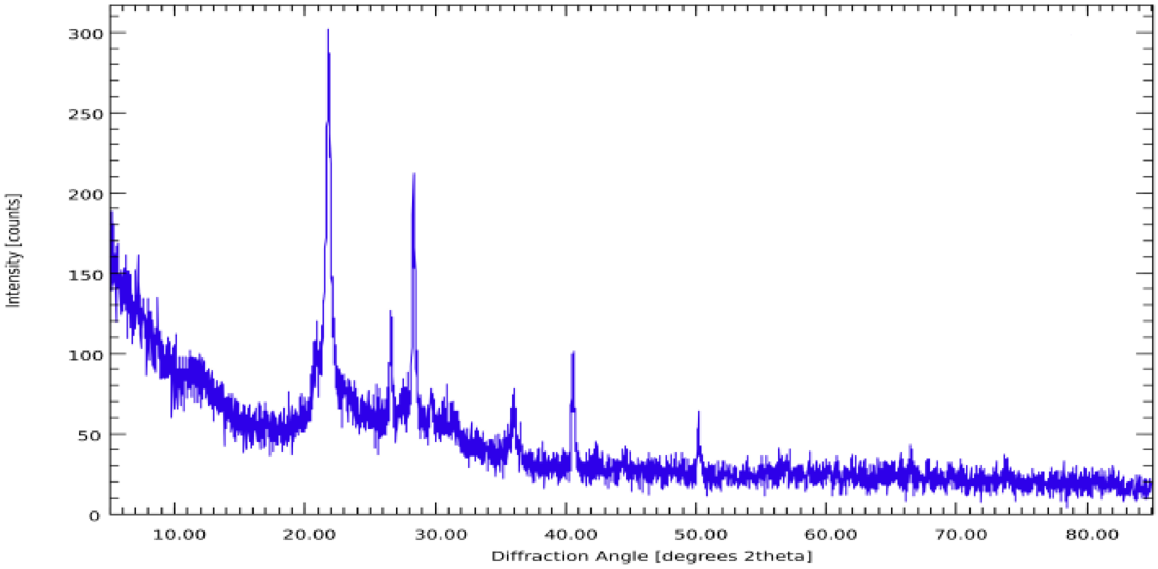


Figure 5: XRD peak distribution for rice straw ash.

XRD analysis serves as a pivotal tool in the investigation and valorisation of crop straw residue in agriculture. It allows for the identification of the crystalline structure inherent in components like cellulose, lignin, and hemicellulose found in crop straw residue. This identification is vital as it provides insights into potential applications and valorisation pathways. Additionally, XRD enables quantification of mineral composition, including essential nutrients like silica, potassium, calcium, and magnesium, informing decisions regarding residue utilization as soil amendments or fertilizers. Moreover, XRD analysis is instrumental in monitoring structural changes during valorisation processes such as pretreatment and enzymatic hydrolysis, facilitating optimization for enhanced biomass conversion efficiency and product quality.

2.3. Electron Probe Microanalysis (EPMA)

EPMA was conducted for quantitative chemical analysis, utilizing a wavelength dispersive spectrometry (WDS) detector for accuracy and precision. In Figure 6, the Backscattered electron (BSE) image reveals a heterogeneous microstructure within the rice straw ash sample. The image exhibits a distinct contrast variation, indicative of compositional heterogeneity. Brighter regions within the image likely correspond to areas enriched in heavier elements, such as silica or metal oxides, commonly found in rice straw ash. Conversely, darker regions suggest a lower atomic number composition, potentially representing carbon-rich or organic residues.

The overall morphology of the ash appears porous and irregular, with a substantial presence of fine particulates.

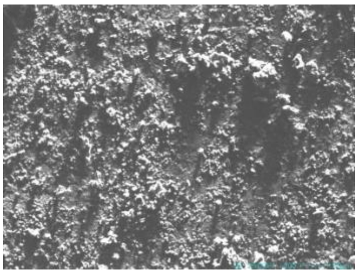


Figure 6: Backscattered electron (BSE) image of sample Rice straw ash.

The EPMA measurements yielded the qualitative distribution of elements carbon (C), nitrogen (N), magnesium (Mg), silicon (Si) and calcium (Ca) across a 600x450 µm^2 area, as illustrated by the intensity maps in Figure-7. The carbon mapping shows a maximum concentration of 49.62 mass%, with an average of 0.39 mass% and variability indicated by a standard deviation (SD) of 2.57 mass%, indicating that while there are areas with high carbon, most of the sample has low carbon content. The nitrogen mapping indicates a maximum of 39.11 mass%, an average of 6.38 mass%, and a high SD of 35.41 mass%, suggesting localized nitrogen accumulation. Magnesium is present at a maximum of 10.95 mass%, with an average of 0.31 mass% and an SD of 0.95 mass%, indicating relatively uniform distribution. The silicon map reveals a significant presence with a maximum of 85.41 mass%, an average of 8.96 mass%, and an SD of 10.06 mass%, highlighting its dominant role in the sample. Calcium shows a maximum of 20.53 mass%, an average of 0.57 mass%, and an SD of 1.34 mass%, indicating a higher maximum concentration of calcium with relatively low variability. These detailed EPMA maps underscore the heterogeneous distribution and variability of elements within the sample.

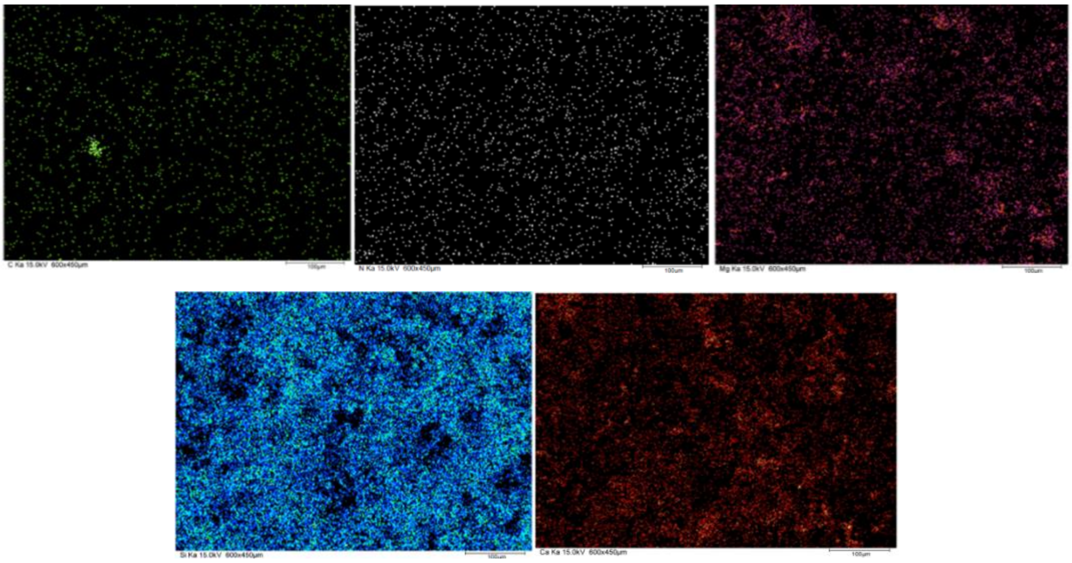


Figure 7: Wavelength-dispersive spectrometry (WDS) intensity map (600x450µm) of C, N, Mg, Si and Ca.

Chemical Composition of the RSA comprises elements with concentration C (0.7%), N (1%), Mg (3.2%), Si (37.4%) and Ca (6%). The chemical composition of RSA is testimony to the promise it holds as a soil amendment agent. RSA, resulting from the combustion of rice straw residues, contains a diverse array of minerals and compounds, in addition to C, N, Mg, Si and Ca, that influence soil properties and nutrient availability positively.

Table 1: Chemical properties of soil in North India

Soil properties	Concentration in soil (%)	References
Soil organic carbon (SOC)	0.51% - 0.62%	Gaur et al. (2023); Sheoran et al. (2019); Pathak et al. (2017)
Nitrogen (N)	52.9-174.05 kg ha^{-1}	Tripathi et al. (2023), Prem et al. (2017)
Magnesium (Mg)	1.80%- 22.55%	Chinchmalatpure et al. (2014)
Calcium Carbonate ($CaCO_3$)	0.61% -4.4%	Mondal and Ramkala (2016); Singh et al. (2014)
Silicon (Si)	20% -30%	Jinger et al. (2020)

In North India, soil properties are very sensitive to the prevalent agricultural practices. For instance, in Karnal, Haryana, where the rice-wheat cropping system predominates, conservation agricultural practices like zero tillage have been adopted in wheat fields. Results show that zero tillage, especially when coupled with residue application, leads to a significant increase in soil organic carbon (SOC) content, reaching up to 0.62% (Table 1). The levels of Mg and N, which are important nutrients for plant growth, vary widely. While the Mg content may fluctuate across different soil types, it generally falls within a moderate range, aiding in enzyme activation, chlorophyll synthesis, and overall plant vigour. However, soil nitrogen levels often present a challenge, with widespread deficiencies observed. This scarcity of nitrogen can limit crop productivity and affect plant development, necessitating strategic fertilization practices to supplement soil nitrogen. Additionally, the ash serves as a valuable source of essential plant nutrients such as calcium and magnesium, gradually releasing them into the soil as it dissolves in moisture. This gradual nutrient release ensures sustained nutrition for plant growth and contributes to overall soil fertility. Carbon and nitrogen are fundamental elements contributing to soil organic matter (SOM) content and nutrient cycling, essential for maintaining soil fertility and supporting plant growth. Mg, Ca, and Mn play crucial roles as micronutrients involved in various metabolic processes within plants, influencing their physiological functions and overall health. Moreover, the fine particles in RSA aid in improving soil structure by enhancing aggregation, water retention, and drainage capacities. By increasing soil cation exchange capacity and stimulating microbial activity, RSA promotes nutrient retention, organic matter decomposition, and nutrient cycling processes in the soil.

2.4. SEM of rice straw

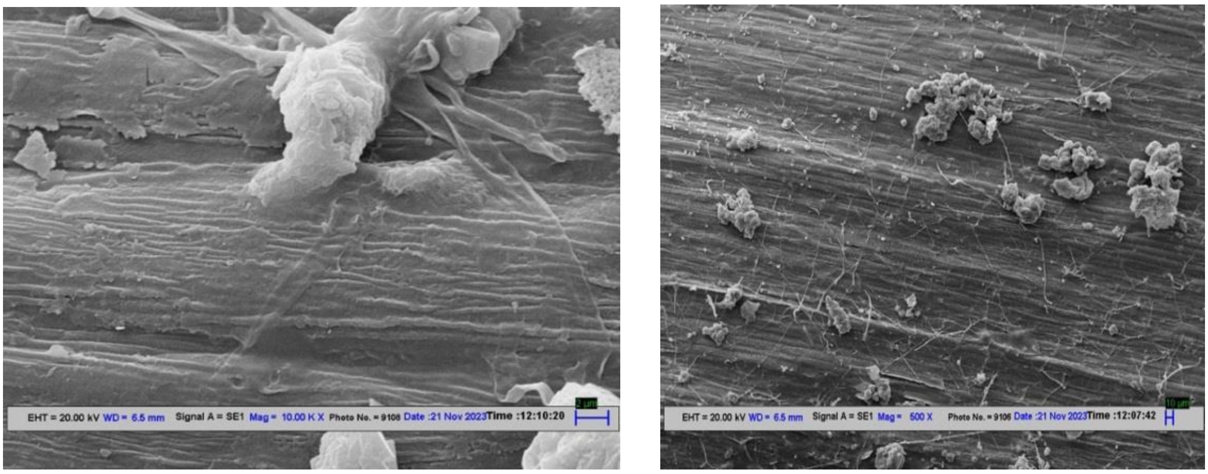
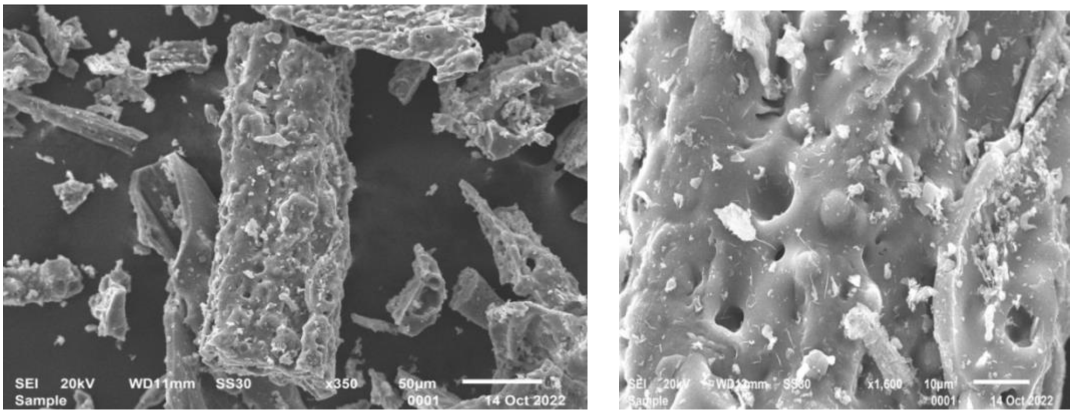


Figure 8: Scanning electron micrograph (SEM) of rice straw at 500x and 10,000x magnification.

A ZEISS EVO Scanning Electron Microscope, operated at 30 kV accelerated voltage, was used to examine the morphology of the rice straw at 500x and 10,000x magnification. The micrograph revealed a structured, firm, and well-arranged composition, as shown in Figure 8. The external surface of the rice straw exhibits clear organization with some areas displaying corrugation. Additionally, the high-resolution images uncover fine spots, known as papillae, and silicaceous structures referred to as phytoliths. These phytoliths appear as sizable clusters on the surface in Figure 8, appearing in the form of sizable clusters (Amido et al., 2021; Kaur et al., 2018; Tsai et al., 2023). At the lower magnification of 500x, the straw's surface appears organized and robust, indicating a well-arranged cellular structure. As magnification increases to 10,000x, finer details become visible, showing that the surface features corrugation or undulating patterns, which suggest textural complexity.

SEM of rice straw ash



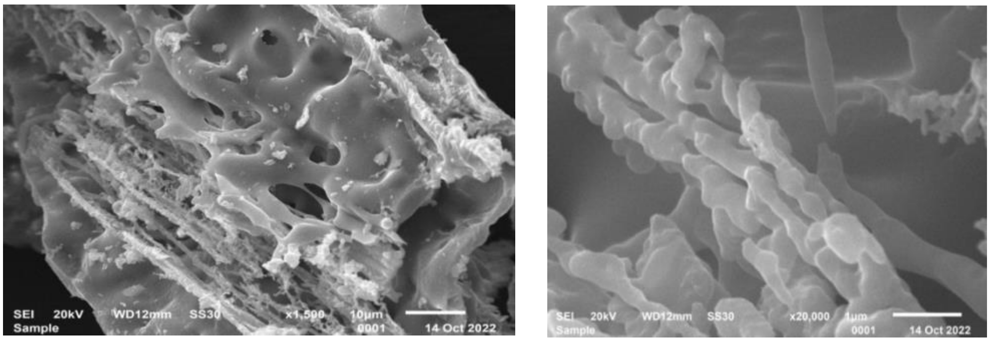


Figure 9: SEM micrographs of RSA.

A JEOL JSM 6610LV Scanning Electron Microscope was used to investigate the morphology of RSA at 350x, 1500x, 1600x and 20,000x and micrographs show the presence of irregular shapes and porous structures [seen in Figure 9, and noted in Ismail et al. (2016) and Amin et al. (2022)].

2.5. Transmission Electron Microscopy (TEM) of rice straw ash

The JEM-1400 Transmission Electron Microscope was used to examine RSA particles. TEM images are presented in Figure 10, at magnifications of 6000x and 8000x. The images portray spherical particles with varied size distributions (Uda et al., 2020), possibly indicating the presence of residual organic matter or inorganic compounds that formed spherical structures during the ashing process. TEM analysis of rice straw ash revealed a complex microstructure characterized by a heterogeneous distribution of particles. Significant portions of the images display an amorphous structure, lacking a distinct crystalline lattice.

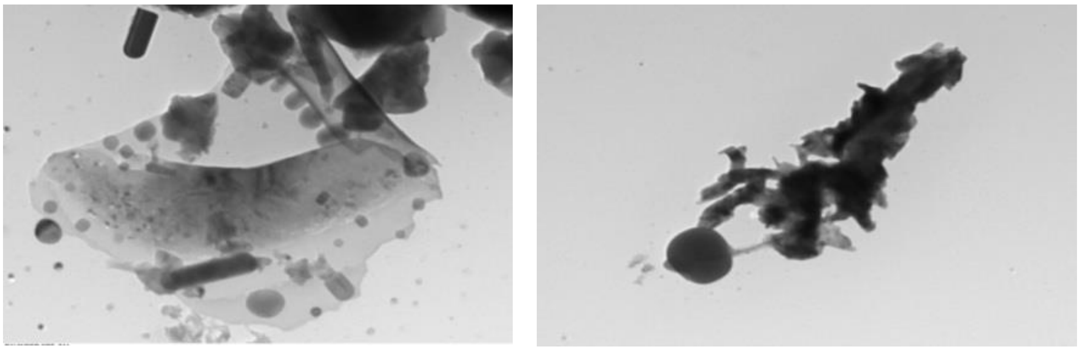


Figure 10: TEM analysis of RSA.

The microscopic structure of RS and RSA observed using SEM and TEM analyses offers critical insights into their morphology, composition, and properties. The presence of both amorphous and crystalline regions was evident, suggesting a diverse composition. Particles exhibited a tendency to agglomerate, forming clusters within the ash matrix. While the amorphous nature of many particles hindered precise identification, potential components based on the typical inorganic constituents of rice straw include silica, which often presents in amorphous or crystalline forms, carbon residues exhibiting variable morphologies, and metal oxides derived from the original plant material. SEM and TEM enable high-resolution imaging of the internal and superficial structures, revealing features such as pores, cracks, and nanostructures within the RS matrix. Understanding the surface morphology and particle size distribution is essential for assessing the accessibility of RS to reactive enzymes and chemicals in biorefinery processes. Additionally, TEM allows for the visualization of nanostructures within RS, like cellulose nanofibrils and lignin nanoparticles, which have implications for the development of nanomaterials and functional biomaterials.

2.6. Dynamic Light Scattering (DLS)

The RSA particle-size distribution was analysed using a Horiba scientific nanoPartica nanoparticle analyser. The DLS analysis was performed with the scattered angle of 90°. The measurement of the specific surface was analysed using RSA (10 mg) mixed in double-distilled water. The particle size range, mean value and standard deviation are 2.4nm to 549.7nm, 245.65 nm, and 44.61 nm respectively. Most particles fell within the size range of 200 to 300 nm.

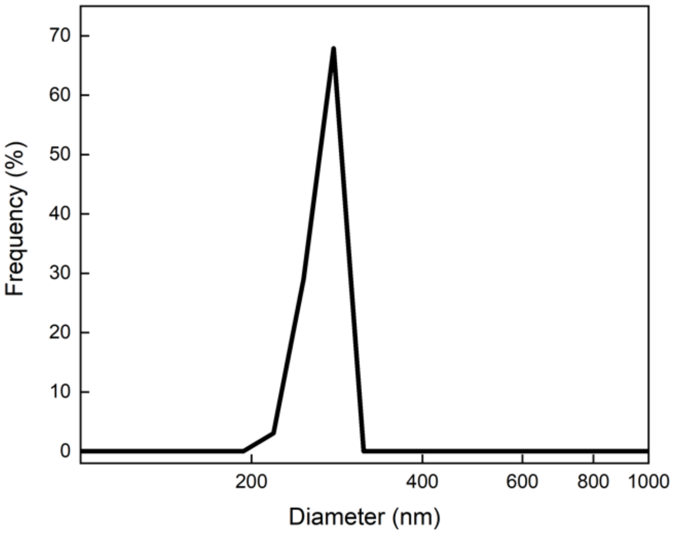


Figure 11: DLS analysis of RSA.

DLS is an indispensable analytical technique in the investigation and valorisation of crop straw residue for agricultural purposes. It facilitates a comprehensive understanding of the particle-size distribution within the RSA. By precisely delineating particle sizes and distributions, this technique aids in grasping the physical attributes of the residue, including the presence of aggregates or colloidal structures. Such insights are paramount for refining valorisation processes, such as pretreatments in soil ensuring thereby, their efficacy in converting crop straw residue into valuable agricultural resources. The technique's ability to monitor changes in particle-size distribution over time enables the researcher to evaluate dispersion stability more accurately. This understanding facilitates the formulation of stable colloidal systems essential for various agricultural applications, ultimately enhancing processing efficiency and product quality.

Conclusion

This study addresses the pressing sustainability challenges posed by the extensive rice-wheat crop rotation system in India, which generates significant crop residues often burned in fields, contributing to greenhouse gas emissions and atmospheric pollutants. By investigating the feasibility of using rice straw ash (RSA) as a soil amendment, this research offers a novel solution to valorize agricultural residues. Utilizing comprehensive analytical techniques such as TGA, XRD, SEM, TEM, and DLS, the study provides critical insights into the thermal behavior, morphology, and mineral composition of RSA. The high silicon (Si) content in RSA indeed makes it a strong candidate for use as a cement additive, primarily because silicon dioxide (SiO_2) is a critical component in the cement hydration process. RSA's silica content can act as a pozzolanic material contributing to improved strength and durability of concrete. This would reduce the need for traditional cement, lowering carbon emissions associated with cement production—a significant contributor to global greenhouse gas emissions. However, comparing the use of RSA as a cement additive versus its valorization in agricultural or environmental applications requires a holistic view. Using RSA as a cement additive helps mitigate air pollution and reduce reliance on conventional cement, aligning with SDG 13 (Climate Action) by lowering CO_2 emissions from cement manufacturing. At the same time, RSA as a soil amendment supports the soil-to-soil circularity paradigm, enhancing soil nutrition, improving water retention, and promoting microbial activity due to its silica content. This practice is beneficial for agricultural sustainability and aligns with SDGs 2 (Zero Hunger), 3 (Good Health and Well-being), and 15 (Life on Land). In terms of direct benefits, cement additive usage may offer a more immediate and large-scale impact on the construction industry by improving material properties and lowering carbon footprints. However, valorization in agriculture ensures the long-term sustainability of agricultural practices, contributing to soil health and promoting a circular bioeconomy. Therefore, the most beneficial use depends on the specific goals—whether it's reducing emissions and improving construction material sustainability or enhancing soil health and supporting sustainable agriculture. In practice, integrating both applications could maximize RSA's value, simultaneously addressing environmental and industrial challenges. Both approaches contribute significantly to sustainability goals, and selecting between the two will depend on regional priorities, availability, and infrastructure. Future studies should focus on optimizing the use of RSA in both agricultural and industrial applications. In agriculture, further research is needed to understand the long-term effects of RSA on different soil types, plant species, and ecosystems. In the construction industry, the efficacy of RSA as a cement additive should be tested under various environmental conditions to determine its viability as a replacement for conventional materials.

Alternative solutions to straw burning, such as the use of biorefineries or mechanical harvesting for direct RSA utilization, should be explored to minimize the harmful impacts of open-field burning. Further research and policy interventions must focus on technologies that reduce emissions during combustion processes and promote cleaner methods of converting rice straw into valuable products without harming the environment. Thus, while the act of burning straw presents environmental challenges, its role in the production of RSA can be justified if these applications lead to substantial long-term benefits, such as enhanced soil health, sustainable construction practices, and broader circular bioeconomy goals.

Author Contributions: Investigation, T.S.; Formal analysis, T.S.; Data curation, T.S.; Writing – original draft, T.S.; Supervision, R.R.C.; Conceptualization, R.R.C.; Methodology, U.S.; Resources, M.S.; Visualization, G.V.; Writing – review and editing, G.V. All authors have read and agreed to the published version of the manuscript.

Funding: This research received no external funding.

Institutional review board statement: Not applicable.

Conflicts of Interest: The authors declare no conflict of interest.

Acknowledgements: The authors would like to express their appreciation to the University Science Instrumentation Centre for granting access to their X-ray Diffraction (XRD) instrument and Scanning Electron Microscopy (SEM) facilities, crucial for conducting this research. Furthermore, heartfelt thanks are extended to the Central Research Facility at the Indian Institute of Technology Delhi (IITD) for generously providing access to their Transmission Electron Microscopy (TEM) and Scanning Electron Microscopy (SEM) equipment. Additionally, the authors acknowledge the support of the Indian Agricultural Research Institute (IARI) for their assistance in facilitating aspects of this study. This study benefitted immensely from their support and resources. The authors would like to express sincere gratitude to the reviewers for their insightful comments and valuable suggestions, which greatly contributed to the improvement of this paper. Additionally, the authors extend their thanks to Manav Sinha for his meticulous proofreading of the final version.

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