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Effect of Pyrolysis Conditions on the Surface Morphology and Composition of Sewage Sludge-Derived Biochar

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ABSTRACT

Sewage sludge-derived biochar offers potential for sustainable waste management and agricultural applications. This study investigates the influence of pyrolysis temperature and retention time on the physicochemical and microstructural properties of biochar derived from sewage sludge. Four biochar samples were produced at 300 °C and 600 °C with residence times of 2 and 4 hours. Proximate analysis was conducted to evaluate moisture content, volatile matter, ash content, and fixed carbon, while surface morphology was assessed using Scanning Electron Microscopy (SEM). Moisture content declined slightly with increased severity (12.43% to ~10.34%), indicating most free water was removed at lower temperatures. Results showed that volatile matter decreased from 18.08% to 8.19% with increasing temperature, while ash content increased from 68.62% to 85.60%. Fixed carbon unexpectedly decreased from 13.30% to 4.22% under higher severity. SEM analysis revealed that even at 600 °C, the biochar surfaces remained smooth and compact with minimal pore development. This behavior is attributed to ash accumulation and the thermally stable mineral content of sewage sludge, which suppresses crack formation during pyrolysis. These findings demonstrate that feedstock composition strongly affects biochar structure and may limit its performance in applications requiring high porosity. However, sludge-derived biochar could still be useful for improving soil bulk density or nutrient retention, provided that harmful components such as heavy metals are adequately treated.

Keywords: Biochar, Proximate Analysis, Pyrolysis Temperature, Retention Time, Scanning Electron Microscopy (SEM).

1. INTRODUCTION

Sewage sludge is a by-product of municipal and industrial wastewater treatment processes. Its generation has increased substantially due to urban population growth, industrialization, and improved sanitation infrastructure [1, 2]. While sewage sludge contains valuable organic matter and nutrients such as nitrogen and phosphorus, its direct disposal poses serious environmental and health hazards. In particular, improper landfilling or open dumping can lead to leachate contamination, odour emission, and pathogen proliferation [3,4]. Therefore, effective management and valorisation of sewage sludge are critical components of sustainable waste treatment systems [2,4].

Conventional sludge disposal methods, such as incineration, land application, and landfilling, face increasing scrutiny due to concerns over greenhouse gas emissions, heavy metal content, and long-term environmental sustainability [2,5]. In this context, thermochemical conversion technologies such as pyrolysis have emerged as attractive alternatives. Pyrolysis not only reduces

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the volume and toxicity of sewage sludge but also generates value-added by-products like bio-oil, syngas, and biochar [6]. Among these, biochar has gained considerable attention for its potential applications in soil amendment, carbon sequestration, and environmental remediation [7, 8].

The production of biochar from sewage sludge offers a promising approach to recycling waste while simultaneously enhancing soil quality and environmental health. Compared to plant-based biomass, sewage sludge-derived biochar tends to have higher ash content, a more complex matrix, and greater concentrations of heavy metals, all of which affect its physicochemical properties [1, 9]. As such, controlling the production parameters like pyrolysis temperature and retention time is essential to tailor the characteristics of biochar for specific applications [5,9].

Pyrolysis is a thermal decomposition process conducted in the absence or limited presence of oxygen, and it plays a pivotal role in determining the structural and chemical properties of the resulting biochar. Two key parameters in this process are pyrolysis temperature and retention time, both of which directly influence the extent of carbonization, devolatilization, and pore development in the biochar matrix. Higher temperatures typically lead to increased aromaticity, surface area, and structural stability, while also reducing the content of volatile matter [10, 11]. Retention time, the duration for which the biomass is held at the target temperature, further affects the thermal degradation and reorganization of organic structures [3, 11].

Despite the growing research on sludge-derived biochar, there remains a limited understanding of how different pyrolysis temperatures and retention time influence its microstructure and morphological features. Most existing studies focus on chemical composition, heavy metal content, and adsorption behavior, with relatively less emphasis on the physical texture and porosity of the material. This gap limits our ability to optimize biochar properties for environmental and agricultural applications.

This study aims to investigate the combined effects of pyrolysis temperature and retention time on the physicochemical and microstructural properties of biochar derived from sewage sludge. Two characterization approaches were employed: proximate analysis to determine moisture content, volatile matter, ash content, and fixed carbon; and Scanning Electron Microscopy (SEM) to examine changes in surface morphology. The findings are expected to contribute to a deeper understanding of sludge-to-biochar conversion processes and contribute to the development of optimized biochar products for sustainable environmental and agricultural applications.

2. MATERIAL AND METHODS

The experimental work was designed to investigate the effects of pyrolysis temperature and retention time on the characteristics of sewage sludge-derived biochar using two primary analytical techniques: proximate analysis and scanning electron microscopy (SEM). Sewage sludge was collected, air-dried, and subjected to pyrolysis under controlled laboratory conditions to produce four biochar samples with varying thermal treatments. The resulting biochar was analyzed to determine its compositional attributes of moisture, volatile matter, ash content, and fixed carbon, and to examine surface morphological changes. These methods were selected to provide insights into the thermal transformation of sewage sludge and to assess the suitability of the resulting biochar for use as a soil amendment.

2.1 Biochar Preparation

Sewage sludge was obtained from one of the Sanitary Solid Waste Disposal Sites located in Rimba Mas, Chuping, Perlis, Malaysia, and served as the feedstock for biochar production. Prior to pyrolysis, the sludge was air-dried for 48 hours and then oven-dried at 105 °C until a constant weight was achieved. Approximately 50 g of the pretreated sludge was placed in a covered ceramic crucible and pyrolyzed in a muffle furnace under atmospheric pressure.

Pyrolysis was conducted at two selected temperatures (300 °C and 600 °C) and two retention times (2 hours and 4 hours), resulting in four treatment combinations. The samples were labeled as follows: Sample 1 – 300 °C for 2 hours, Sample 2 – 300 °C for 4 hours, Sample 3 – 600 °C for 2 hours, and Sample 4 – 600 °C for 4 hours. These conditions were chosen to represent low and high thermal severity, enabling comparisons of their effects on the structural and compositional properties of the biochar. The furnace was preheated to the target temperature before sample introduction. Each pyrolysis condition was carried out in replicate to ensure reproducibility. After pyrolysis, the furnace was allowed to cool to ambient temperature, and the resulting biochar was collected, weighed, and stored in airtight containers for subsequent analysis.

2.2 Proximate Analysis

Proximate analysis was performed to determine the moisture content, volatile matter, ash content and fixed carbon of the biochar samples, following standard procedures adapted from Standard Method 2540E for sludge [12]. All values were calculated on a weight percentage basis (wt%), as commonly applied in compositional analysis of biochar. All proximate analyses were performed in replicate, and results are reported as average values. These measurements were used to evaluate the degree of carbonization and mineral retention under varying pyrolysis conditions.

Moisture content was measured using the MX-50 moisture analyzer. It operates based on thermogravimetric principles by heating the sample with a halogen lamp and continuously recording the weight loss until a stable reading is achieved [13,14]. Approximately 1 g of each biochar sample was used, and the moisture content was calculated automatically by the instrument.

Volatile matter was determined by placing air-dried samples in a covered crucible and heating them at 550 °C for 10 minutes in a muffle furnace. The percentage of volatile matter was calculated using the formula:

$$Volatile\ Matter,\ wt\ \% = \frac{Weight\ of\ sample\ before\ drying - Weight\ of\ sample\ after\ drying}{Weight\ of\ sample\ before\ drying} \tag{1}$$

Ash content was measured by heating the samples in an uncovered crucible at 400 °C for 4 hours. The remaining residue was recorded as ash content, calculated using the formula:

$$Ash\ content, wt\ \% = \frac{Weight\ of\ residue}{Weight\ of\ sample} \tag{2}$$

Fixed carbon was calculated by difference, using the following equation:

$$Fixed\ Carbon, wt\ \% = 100 - [\ ash\ (wt\ \%) + volatile\ matter\ (wt\ \%)] \tag{3}$$

2.3 Scanning Electron Microscopy (SEM)

Surface morphology of the biochar samples was analyzed using a TESCAN VEGA 4th-generation scanning electron microscope (SEM), equipped with a tungsten filament electron source. SEM is one of the most widely used techniques for surface characterization in biochar research [15-17]. The instrument integrates high-resolution SEM imaging with live elemental analysis through TESCAN's Essence™ software, enabling simultaneous acquisition of morphological and compositional data within a unified interface. Imaging was performed under high-vacuum mode at an accelerating voltage of 10–15 kV, with magnifications ranging from 100× to 10,000×. Prior to imaging, the samples were mounted on carbon adhesive tape and sputter-coated with a thin layer of gold to enhance conductivity and minimize charging effects. The SEM micrographs were used to assess surface texture, evidence of pore development, and structural responses to varying pyrolysis conditions. The observations were qualitative in nature, and no pore size distribution or image analysis was performed.

3. RESULTS AND DISCUSSION

This section presents the effects of pyrolysis temperature and retention time on the properties of sewage sludge-derived biochar. It is divided into two parts: Section 3.1 discusses the basic composition of the biochar based on proximate analysis, while Section 3.2 describes the surface structure observed through Scanning Electron Microscopy (SEM). Together, these results help explain how heat treatment affects both the chemical content and physical appearance of the biochar.

3.1 Physicochemical Characteristics of Biochar

The proximate analysis results of the four biochar samples are presented in Table 1. These results indicate that pyrolysis temperature and residence time significantly influence the composition of sewage sludge-derived biochar. The key parameters analyzed include moisture content, volatile matter, ash content, and fixed carbon.

Table 1: Proximate composition of biochar samples produced at varying pyrolysis temperatures and retention times.

Sample	Treatment	Moisture (%)	Volatile Matter (%)	Ash Content (%)	Fixed Carbon (%)
1	300°C, 2 hr	12.43	18.08	68.62	13.30
2	300°C, 4 hr	10.02	17.05	72.11	10.84
3	600°C, 2 hr	10.40	8.19	83.20	8.53
4	600°C, 4 hr	10.34	10.20	85.60	4.22

As shown in Table 1, the moisture content was highest in the sample pyrolyzed at 300 °C for 2 hours (12.43%) and generally decreased with increased retention time or temperature. However, the differences in moisture content between samples 2 to 4 were relatively small, indicating that most free water was removed at lower temperatures, with diminishing returns at longer or higher severity pyrolysis. Similar behavior has been observed in sewage sludge biochar, where moisture content significantly decreases with increasing pyrolysis temperature due to enhanced drying efficiency [18].

Volatile matter (VM) showed a substantial decrease with increasing pyrolysis temperature from 300 °C to 600 °C. Samples 1 and 2 (300 °C) exhibited higher volatile contents compared to Samples 3 and 4 (600 °C), which showed significantly lower values. This trend reflects the more complete breakdown of labile organic compounds at higher pyrolysis temperatures. These results align with studies reporting that elevated pyrolysis severity leads to greater devolatilization and significantly reduced volatile matter in sewage sludge-derived biochar [3,9].

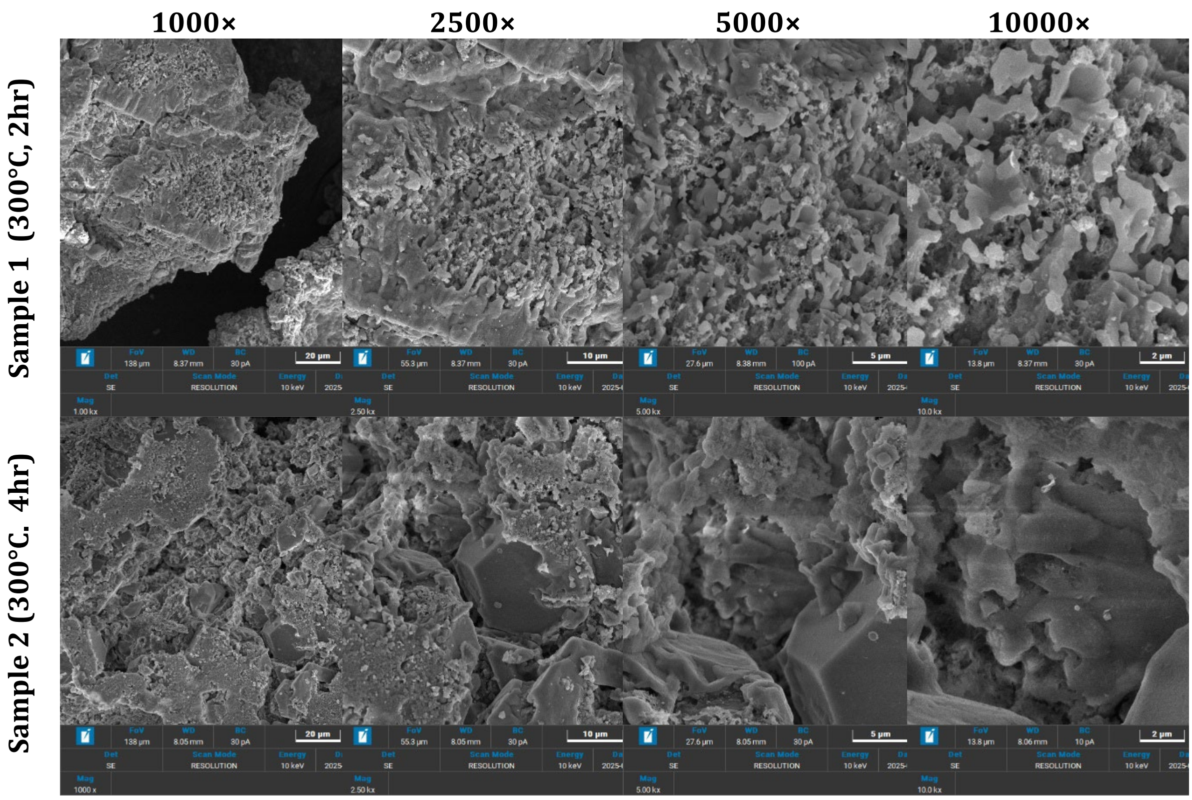
In contrast, ash content increased with both temperature and time. The lowest ash content was observed in Sample 1 (68.62%), while the highest was in Sample 4 (85.60%). This accumulation of ash is expected, as mineral components become concentrated due to the loss of organic matter during pyrolysis. These findings are consistent with Souza et al., who reported increased ash levels in biochar produced at higher pyrolysis temperatures [9].

At 300 °C, fixed carbon decreased from 13.30% (2 h) to 10.84% (4 h), while at 600 °C it dropped further from 8.53% to 4.22%. Fixed carbon was consistently higher at 300 °C than at 600 °C. This may be due to carbon breakdown or oxidation during prolonged heating at high temperatures, especially at 600 °C for 4 hours. This suggests that very high temperatures and longer retention times can reduce stable carbon, particularly if the system is not fully airtight, leading to oxidation during pyrolysis. Souza et al. also observed a decline in fixed carbon with increasing temperature [9].

In summary, increasing the pyrolysis temperature from 300 °C to 600 °C led to lower volatile matter, higher ash content, a slight reduction in moisture, and an unexpected decrease in fixed carbon. Similarly, extending the retention time from 2 h to 4 h resulted in further reductions in moisture and volatile matter, along with increased ash and reduced fixed carbon. These findings highlight the combined influence of temperature and time on the physicochemical composition of sewage sludge-derived biochar. Higher temperatures and longer heating times promote devolatilization and concentrate mineral content. However, they can also reduce the amount of stable carbon, which may affect the quality of biochar for use in soil or carbon storage applications.

3.2 Microstructural Analysis of Biochar

The surface morphology of the biochar samples was examined using Scanning Electron Microscopy (SEM) at various magnifications ranging from 1000× to 10,000× (Figure 1). The micrographs reveal clear differences in surface structure across samples produced under different pyrolysis conditions, indicating that both temperature and retention time strongly influence the physical characteristics of biochar.



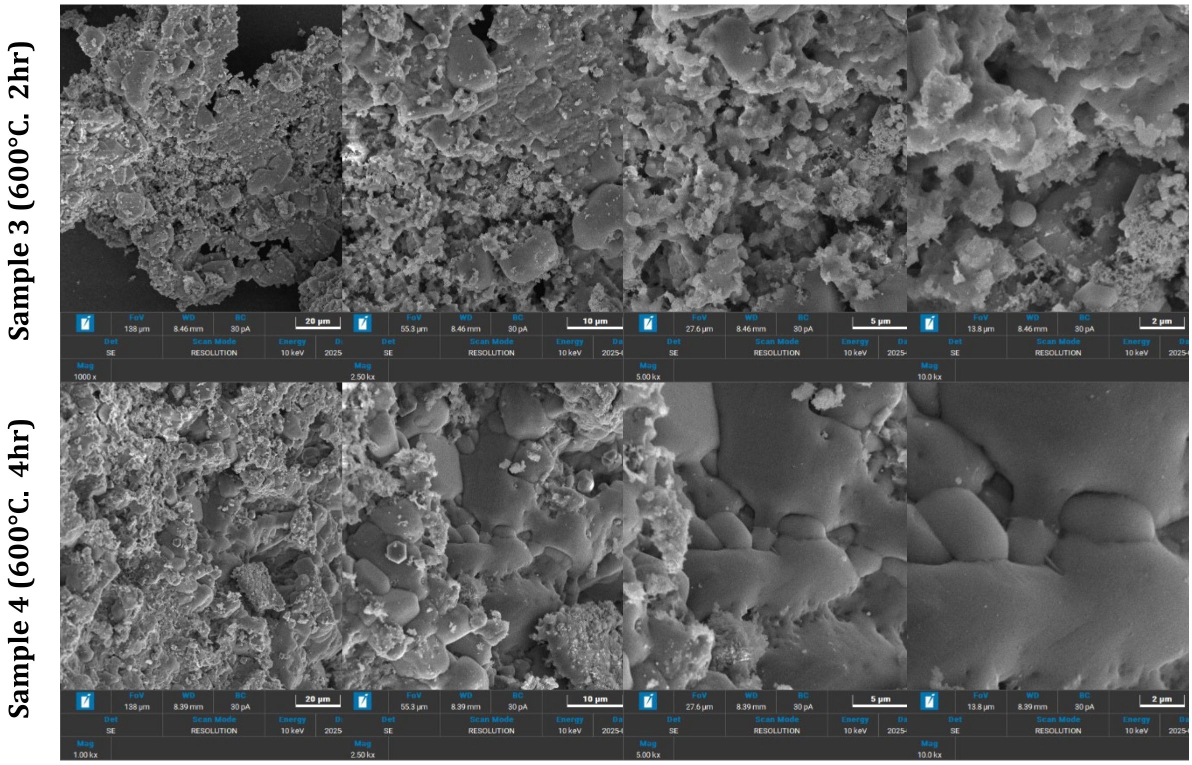


Figure 1: SEM micrographs of sewage sludge-derived biochar samples at varying pyrolysis conditions and magnifications. (a–d) Sample 1, (e–h) Sample 2, (i–l) Sample 3, and (m–p) Sample 4, each shown at magnifications of 1000×, 2500×, 5000×, and 10,000×, respectively.

At 300 °C (Samples 1 and 2), SEM micrographs revealed smooth, compact surfaces with only minor roughening at higher magnifications. Extending the residence time led to slightly more surface irregularity, suggesting partial transformation. However, significant pore networks were still absent. This reflects limited devolatilization at lower temperatures and aligns with observations by Halalsheh et al., who reported minimal morphological evolution at low pyrolysis severity in sludge-derived biochar [3].

At 600 °C (Samples 3 and 4), microcracking and textural changes became more apparent, particularly for Sample 3. Yet even under these conditions, many regions remained smooth and dense rather than porous. This persistent lack of porosity, despite high thermal severity, likely stems from the dominance of mineral residues in the biochar. Tian et al. found that sludge biochar rich in ash, particularly kaolin, metakaolin, and silicates, tends to mask the surface structure and suppress texture development. This limits both adsorption and visible pore formation [19]. Studies examining sewage sludge biochar morphology have confirmed that high inorganic content can result in fused, nonporous surfaces even at elevated temperatures [20].

The differences observed in this study can be attributed to the nature of the feedstock, sewage sludge, a by-product of municipal and industrial wastewater treatment processes, known to be rich in inorganic mineral residues. These inorganic minerals, such as silicates, phosphates, and metal oxides, are thermally stable and may melt or sinter at elevated temperatures, coating the biochar surface and suppressing the formation or visibility of fine cracks and pores. As a result, the overall morphology remains compact, with limited internal voids, even at 10,000× magnification. This effect is characteristic of sludge-derived biochar and differs significantly from biomass-based feedstocks with higher organic content [19, 21-22].

In summary, the SEM results confirm that pyrolysis temperature and retention time do influence surface morphology, but their effects are strongly mediated by the chemical composition of the feedstock. The limited pore development, dense structure, and high ash content observed in sludge-derived biochar may reduce its suitability for applications requiring high surface area, such as water retention or nutrient adsorption. However, these characteristics can still offer benefits in specific soil contexts, such as improving bulk density or reducing nutrient leaching. For use as a soil amendment, further treatment may be necessary to remove or stabilize potentially harmful substances, including heavy metals, to ensure environmental safety. These findings are important for understanding both the structural limitations and potential functionalities of sludge-derived biochar, particularly when designing post-treatment strategies to enhance its application potential in agriculture and environmental remediation.

Heavy metals are a critical consideration for sludge-derived biochar, as elements such as Cd, Pb, Zn, and Cu may become concentrated during pyrolysis. Although not measured in this study, their potential mobility must be evaluated to ensure safe agricultural use. Future work should include leaching tests, nutrient retention studies, and field trials to validate agronomic benefits and guide practical utilisation.

4. CONCLUSION

This study aimed to evaluate the influence of pyrolysis temperature and retention time on the physicochemical and microstructural properties of sewage sludge-derived biochar. The results showed that higher pyrolysis temperatures and longer retention times reduced moisture and volatile matter while increasing ash content, although fixed carbon unexpectedly decreased under more severe conditions. SEM analysis revealed that the biochar surfaces remained compact with limited pore development, primarily due to the high inorganic content and ash accumulation typical of sewage sludge feedstock. These findings suggest that although sewage sludge can be converted into stable carbon-rich material, its structural features may limit applications that require high surface area or porosity. Future research should explore strategies such as feedstock blending or post-treatment to enhance its functionality for environmental and agricultural use.

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