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To cite this article: Nimas Mayang Sabrina Sunyoto *et al* 2026 *IOP Conf. Ser.: Earth Environ. Sci.* **1617** 012017

IOP Conf. Series: Earth and Environmental Science **1617** (2026) 012017 doi:10.1088/1755-1315/1617/1/012017

Effect of H_2SO_4 solution concentration and pyrolysis temperature on the characteristics of activated micro biochar from sugarcane bagasse waste

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Abstract. Sugarcane (*Saccharum officinarum L.*) is the main commodity used as raw material for sugar production. National sugarcane production reached 2,271,000 tons in 2023, with East Java as the largest producing province. One of the main wastes from sugarcane milling is bagasse, which accounts for 35-40% of the total weight of fresh sugarcane. Bagasse waste contains 40-50% cellulose, 20-30% hemicellulose, and 10-25% lignin, giving high potential for utilization, one of which is as raw material for activated micro biochar. This study aims to analyze and evaluate the effect of pyrolysis temperature and concentration of H_2SO_4 solution on the characteristics of activated micro biochar from bagasse waste. The study used two factors: pyrolysis temperature with three levels (400°C, 500°C, and 600°C) and H_2SO_4 concentration with three levels (1M, 3M, and 5M). The results showed that pyrolysis temperature significantly affected all test parameters, while the concentration of H_2SO_4 solution also influenced all parameters. The best treatment was activated micro biochar produced at a pyrolysis temperature of 600°C and H_2SO_4 concentration of 5M. This treatment met three of the six parameters of SNI 06-3730-1995 regarding quality requirements and testing of activated carbon, with characteristics of moisture content 2.20%, ash content 13.63%, volatile matter 19.87%, fixed carbon 66.50%, iodine adsorption 721.55 mg/g, methylene blue adsorption 17.94 mg/g with 92.03% efficiency, and particle size 13.604 μm. This research supports the potential application of activated micro biochar in wastewater adsorption based on its physicochemical properties.

1. Introduction

Sugarcane (*Saccharum officinarum L.*) is one of the main plantation commodities in Indonesia and is widely used as raw material for sugar production. In 2023, national sugarcane production reached 2,271,000 tons, with East Java contributing the highest amount at 1,129,400 tons [1]. The sugar milling process generates bagasse as its main solid waste, accounting for approximately 35–40% of the total weight of fresh sugarcane [2]. Bagasse contains high levels of cellulose (26–

43%) and hemicellulose (17–23%) [3], making it a promising feedstock for the production of biochar, especially activated micro biochar. Activated biochar is increasingly explored due to its ability to adsorb pollutants, porous structure, high surface area, and abundant surface functional groups. Biochar can be further optimized through chemical activation to enhance its physicochemical properties. One of the commonly used activating agents is sulfuric acid (H_2SO_4), which can improve surface area, porosity, and adsorption performance [4]. Sulfuric acid promotes dehydration and sulfonation reactions that increase adsorption capacity [5]. In particular, activated micro biochar with micro scale particle size (<100 μm) has been reported to be more efficient in removing pollutants from liquid waste [6].

Previous studies have investigated the effect of pyrolysis temperature and H_2SO_4 concentration on biochar characteristics. For instance, Savou et al. [7] studied sugarcane bagasse activated with H_2SO_4 concentrations of 1M, 3M, 6M, and 9M at 500°C and found the optimal condition at 3M, yielding 13.4 wt% tar and 31.6% carbon content. Siregar et al. [8] applied pyrolysis at 300°C for 2.5 hours with 1M H_2SO_4 and produced biochar with 5.9% moisture content, 5.6% volatile matter, 1.4% ash, and 58% water adsorption. Similarly, Sohaib et al. [9] showed that a pyrolysis temperature of 600°C gave the best result, with 2.97% moisture, 7.58% ash, 20.35% volatile matter, and 69.10% fixed carbon. These findings highlight that pyrolysis temperature and activator concentration are key factors that determine the quality and performance of biochar. In Indonesia, biochar quality is also benchmarked against the national standard SNI 06-3730-1995, which sets parameters such as moisture, ash content, volatile matter, and fixed carbon for activated carbon. Among its environmental applications, activated micro biochar has demonstrated strong potential for removing hexavalent chromium (Cr(VI)), a toxic and carcinogenic heavy metal found in industrial wastewater. The presence of oxygen-containing functional groups and enhanced porosity makes it effective for Cr(VI) adsorption, positioning it as a promising alternative in wastewater treatment technologies [10]. Specific studies on activated micro biochar derived from sugarcane bagasse, especially involving variation in pyrolysis temperature and H_2SO_4 concentration, remain limited. Therefore, this study aims to analyze the effect of pyrolysis temperature and sulfuric acid concentration on the physicochemical characteristics of activated micro biochar from sugarcane bagasse. The research is expected to support its application in environmental adsorption and waste water treatment.

2. Materials and Methods

The research was conducted from December 2024 to May 2025. Activation, characterization, and testing of the activated micro biochar were carried out at the Agroindustrial Process Engineering Laboratory, Bioindustry Laboratory, and Integrated Laboratory, Faculty of Agricultural Technology, Universitas Brawijaya. The pyrolysis process was conducted at the Renewable Energy Laboratory, Universitas Tribhuwana Tunggadewi Malang. The tools used in this study included a fixed-bed pyrolysis reactor (300 g capacity), ball mill, blender, analytical balance, Memmert UN55 oven, hot plate stirrer, Nabertherm furnace, orbital shaker, desiccator, 200-mesh sieve, porcelain crucibles and dishes, clamps, beakers, Erlenmeyer flasks, funnels, measuring cylinders, titration apparatus, UV-Vis spectrophotometer (UV-752N), FTIR spectrometer (QATR-S), particle size analyzer (Shimadzu SALD-7500 nano), and spatulas. The materials used consisted of sugarcane bagasse, distilled water, sulfuric acid (H_2SO_4), 0.1 N iodine solution, 0.25% methylene blue solution, 0.1 N sodium thiosulfate ($Na_2S_2O_3$), along with filter paper, labeling paper, plastic zip bags, and tissue.

This study used a 1:5 (b/v) ratio of micro biochar to H_2SO_4 solution for chemical activation, with bagasse waste sourced from Kebon Agung Sugar Factory, Malang, East Java. Analyses included moisture content, ash, volatile matter, fixed carbon, iodine and methylene blue adsorption, yield, and surface area estimation. The best treatment was selected based on key quality parameters following SNI 06-3730-1995. Particle Size Analyzer (PSA) testing was conducted only on the best sample to confirm its micro-scale size, while FTIR analysis was used to identify functional groups. This study employed a Randomized Block Design with two factors: Factor A was pyrolysis temperature (400°C, 500°C, and 600°C), and Factor B was H_2SO_4 solution concentration (1M, 3M, and 5M). The combination of these levels resulted in 9 treatment groups, each replicated three times, yielding a total of 27 experimental units. A negative control consisting of micro biochar pyrolyzed at 400°C, 500°C, and 600°C without activation was used for comparison. The observed parameters included moisture content, ash content, volatile matter, fixed carbon, iodine number, and methylene blue adsorption, assessed according to SNI 06-3730-1995.

Micro biochar preparation comprises two principal steps, thermal decomposition of sugarcane bagasse via pyrolysis and subsequent particle size reduction using a ball mill. Micro biochar preparation consisted of two main stages, thermal decomposition of sugarcane bagasse through pyrolysis and particle size reduction using a ball mill. The pyrolysis procedure was adapted from Setyawan et al. [11], particularly the use of a fixed-bed reactor and the temperature variations applied (400°C, 500°C, and 600°C). Prior to pyrolysis, sugarcane bagasse was sun-dried for 24 hours, cut into pieces smaller than 4 cm, and weighed to 200 g for each batch. Pyrolysis was then carried out for 1 hour in the fixed-bed reactor under the specified temperatures to produce biochar. The generated biochar was further processed into micro biochar using a ball mill following the modified approaches described in Setyawan et al. [11] and Pamungkas [12]. The biochar was ground using a ball mill at 100 rpm for 2 hours and sieved through a 200-mesh screen to obtain uniform micro-scale particles suitable for further activation and analysis. The activation process of micro biochar was carried out through a modified chemical method. In this procedure, 20 grams of micro biochar were immersed in sulfuric acid (H_2SO_4) solutions at varying concentrations (1M, 3M, and 5M) for 24 hours at ambient temperature. Following the activation, the biochar was filtered, thoroughly washed with distilled water and 0.05 N NaOH until reaching a neutral pH, then re-filtered and oven-dried at 110°C for 3 hours. The activated micro biochar obtained was subsequently evaluated for key physicochemical properties based on SNI 06-3730-1995, including moisture content, ash content, volatile matter, fixed carbon, iodine adsorption capacity, and methylene blue adsorption.

3. Results and Discussion

3.1 Characteristics of micro biochar from bagasse waste

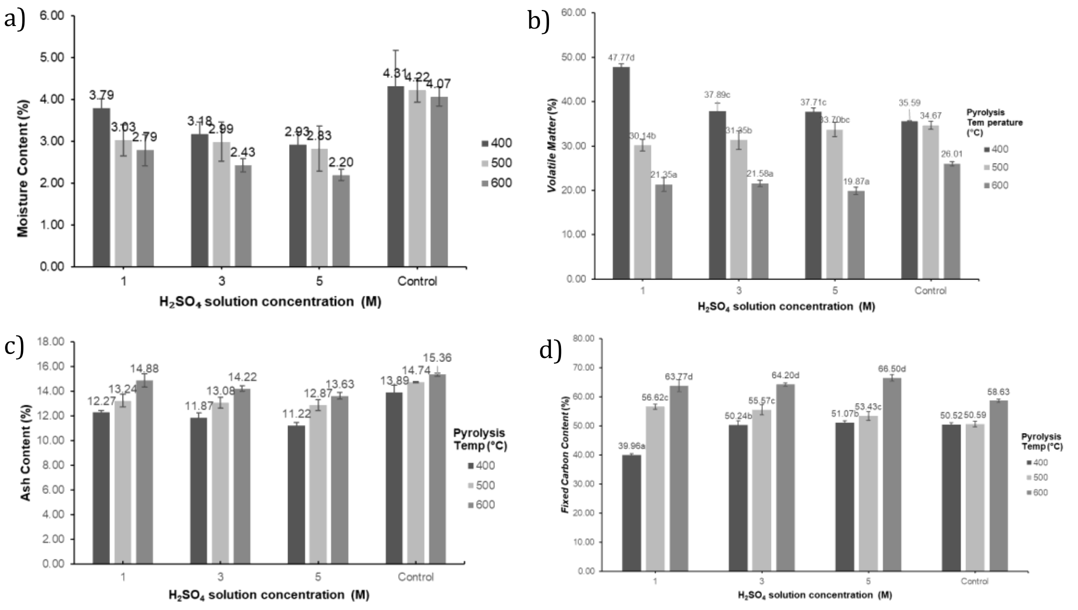
Sugarcane bagasse underwent pyrolysis at 400°C, 500°C, and 600°C for one hour to produce micro biochar, which was then ground using a ball mill and sieved through a 200-mesh screen to obtain a fine, dry, black powder with a characteristic charcoal odor. This material served as the control sample for further analysis. The characterization of micro biochar followed the SNI 06-3730-1995 standard to determine its baseline physicochemical properties, including moisture content, volatile matter, ash content, fixed carbon, iodine adsorption, and methylene blue adsorption. The results in Table 1 indicated that increasing the pyrolysis temperature slightly decreased the moisture content (4.31–4.07 %) and volatile matter (35.59–26.01 %), while increasing the ash content (13.88–15.36 %) and fixed carbon (50.52–58.63 %). However, only the moisture content met the SNI requirement of below 15 %, whereas volatile matter, ash, fixed carbon, iodine number (427.32–543.29 mg/g), and methylene blue adsorption (16.97–17.91 mg/g) did not reach the specified standards. These variations were strongly influenced by the pyrolysis temperature, where higher temperatures promoted thermal decomposition, thereby reducing residual moisture and volatile compounds but simultaneously increasing mineral concentration in the ash fraction. According to Setyawan et al. [11], higher pyrolysis temperatures reduce moisture and volatile matter due to increased thermal decomposition.

Table 1. Characteristics of micro biochar from bagasse waste

Parameters	Results			SNI 06-3730-1995
	400°C	500°C	600°C	
Moisture Content (%)	4.31 ± 0.86	4.21 ± 0.29	4.07 ± 0.23	Max. 15%
Volatile Matter (%)	35.59 ± 0.18	34.67 ± 1.59	26.01 ± 0.84	Max. 25%
Ash Content (%)	13.88 ± 1.03	14.74 ± 0.07	15.36 ± 0.22	Max. 10%
Fixed Carbon (%)	50.52 ± 0.93	50.59 ± 1.61	58.63 ± 0.89	Min. 65%
Iodine Number (mg/g)	427.32 ± 5.01	498.18 ± 1.56	543.29 ± 6.42	Min. 750 mg/g
Methylene Blue Adsorption (mg/g)	16.97 ± 0.67	17.91 ± 0.52	17.26 ± 0.50	Min. 120 mg/g

3.2 Characteristics of activated micro biochar from bagasse waste

The physicochemical characteristics of activated micro biochar from sugarcane bagasse were strongly affected by the combined influence of pyrolysis temperature and sulfuric acid (H_2SO_4) concentration. The moisture content ranged from 2.19 % to 3.78 %, all sample meeting the SNI 06-3730-1995 standard maximum of 15 %. The lowest value was recorded at 600 °C with 5 M H_2SO_4, while the highest occurred at 400 °C and 1 M H_2SO_4. Moisture content plays a crucial role in determining the adsorption efficiency of biochar; lower values generally correspond to improved pore accessibility and enhanced adsorption performance [13]. The significant reduction in moisture at higher pyrolysis temperatures is attributed to the thermal degradation of hemicellulose and lignin, which releases bound water and volatile compounds. Furthermore, the strong dehydrating nature of H_2SO_4 promotes water removal through the formation of sulfated intermediates, facilitating pore development and structural dryness [14,15].



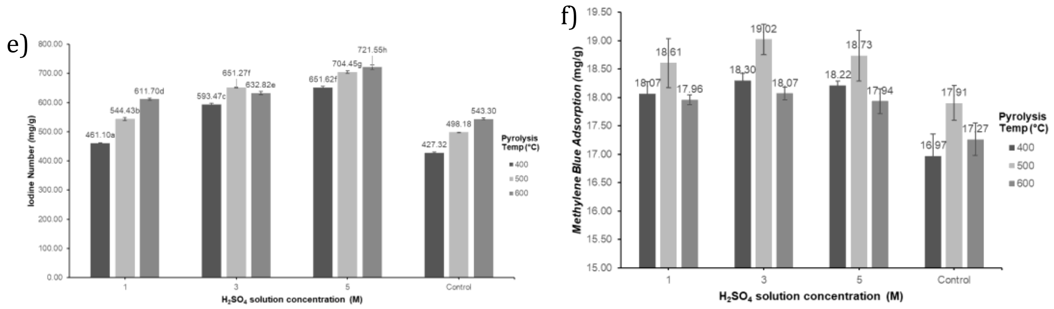


Figure 1. Interaction of pyrolysis temperature and H_2SO_4 concentration on a) moisture content; b) volatile matter; c) ash content; d) fixed carbon; e) iodine number; f) methylene blue

Volatile matter, which represents thermally unstable components, ranged from 47.77% to 19.87%. Samples produced at 600 °C with 5 M H_2SO_4 exhibited the lowest volatile fraction, satisfying the SNI maximum limit of 25 %. A decreasing trend in volatile matter indicates progressive carbonization and improved thermal stability of the biochar structure [16]. Two-way ANOVA results confirmed that both temperature and acid concentration significantly affected volatile matter content ($p < 0.05$), with their interaction also being significant. Higher temperatures enhance devolatilization through the release of light gases such as CO_2 and CH_4, while chemical activation promotes the breakdown of unstable organics, resulting in a more carbon-rich and stable matrix [17]. These findings align with those reported by Sohaib et al. [9], observed similar reductions in volatile matter with increasing pyrolysis temperature. The effect of thermal and acid activation enhances the overall structural order, producing biochar with superior adsorption properties.

Ash and fixed carbon content reflect the inorganic and stable carbon fractions of the biochar, respectively. The ash content ranged from 11.22 % to 16.63 %, exceeding the SNI standard of 10%. The increase in ash with temperature is caused by the accumulation of non-volatile mineral residues after the combustion of organic matter [18,19]. However, acid activation partially counteracted this effect by dissolving inorganic salts and transforming them into water-soluble sulfates that were removed during washing [7]. The fixed carbon content varied from 39.96 % to 66.50 %, achieving the SNI minimum requirement (65 %) only at 600 °C and 5 M H_2SO_4. This increase is attributed to enhanced aromatic condensation and volatilization of oxygenated compounds, resulting in a more thermally stable and graphitic structure [20]. The inverse correlation among volatile matter, ash, and fixed carbon suggests that greater carbonization leads to lower impurities and higher carbon retention, which is essential for developing an efficient adsorbent material.

Surface-related parameters, including the iodine number, methylene blue adsorption, and surface area, provide insight into the textural and adsorption properties of the activated micro biochar. The iodine number increased from 461.10 mg/g at 400 °C–1 M H_2SO_4 to 721.55 mg/g at 600 °C–5 M H_2SO_4, reflecting a substantial improvement in microporosity and surface accessibility [4]. The corresponding surface area values ranged from 508.50 m^2/g to 795.73 m^2/g, confirming that higher temperature and acid strength enhance pore formation through oxidation and dehydration reactions [21]. In contrast, methylene blue adsorption remained low (17.94–19.02 mg/g), indicating that mesopore formation was limited, as strong acid activation predominantly generates micropores less suitable for larger dye molecules [6,22, 23]. Pyrolysis yield decreased from 37.33 % to 31.67 % with increasing temperature, while activation yield ranged between 88.69 % and 95.41 %, with the highest at 600 °C and 1 M H_2SO_4, showing that higher temperature improved carbon stability and minimized mass loss during acid treatment [24,25]. Overall, the combination of elevated pyrolysis temperature and optimal acid concentration significantly enhanced the carbon structure, surface area, and microporosity of the activated micro biochar, making it a promising adsorbent for environmental adsorption and industrial applications.

3.3 Determination of the best treatment

The optimization of activated micro biochar performance was conducted using the Multiple Attribute Zeleny method, which allows multi-parameter evaluation by integrating both desirable and undesirable attributes into a single composite index. The assessment considered six parameters, moisture, volatile matter, ash, fixed carbon, iodine number, and methylene blue adsorption, using the standards specified by SNI 06-3730-1995 as the ideal reference. For optimization, lower values were preferred for parameters such as moisture, ash, and volatile matter, while higher values were considered ideal for fixed carbon, iodine number, and methylene blue adsorption. Based on the composite scores (L1, L2, and L∞ distances), the treatment combination of a 600 °C pyrolysis temperature and 5 M H_2SO_4 activation (A3B3) exhibited the lowest total distance (L1 = 0.038974; L2 = 0.000958; L∞ = 0.029447), indicating its closeness to the ideal condition and confirming it as the best treatment in Table 2. These results demonstrate that synergistic optimization of thermal and chemical activation parameters enhances pore development, stability, and adsorption performance of the resulting micro biochar [14,16].

The A3B3 treatment successfully fulfilled several SNI 06-3730-1995 quality standards, including moisture content (2.20 %), volatile matter (19.87 %), and fixed carbon (66.50 %), confirming effective dehydration, devolatilization, and carbonization at high temperature and acid concentration. However, the ash content (13.63 %) exceeded the maximum limit, which can be attributed to the concentration of inorganic minerals such as silica and calcium and the presence of sulfate residues from the acid treatment [20]. While the iodine number (721.55 mg/g) approached the required minimum of 750 mg/g, it remained slightly lower than that achieved using HCl activation (816.41 mg/g), as reported by Imani et al. [26], suggesting that the type of activating agent significantly affects surface oxidation and pore chemistry. Methylene blue adsorption (17.94 mg/g) was comparable to that obtained by Choi et al. [27], indicating limited mesoporosity formation under sulfuric acid activation. These results emphasize that, although H_2SO_4 activation effectively enhances micropore development, further modification, such as dual activation or alkali-assisted treatment, is necessary to improve mesopore formation for large-molecule adsorption [22].

Table 2. Results of activated micro biochar bagasse waste best treatment

Parameters	Research Result	SNI Standard	Results from Other Research	Sources
Moisture content (%)	2.20	Max 15%	2.97	[9]
Volatile matter (%)	19.87	Max 25%	20.35	[9]
Ash content (%)	13.63	Max 10%	7.58	[9]
Fixed carbon (%)	66.50	Min 65%	69.10	[9]
Iodine number (mg/g)	721.55	Min 750 mg/g	816.41	[26]
Methylene blue adsorption (mg/g)	17.94	Min 120 mg/g	17.48	[27]

The best-performing activated micro biochar (A3B3), obtained from pyrolysis at 600 °C and activation with 5 M H_2SO_4, was further examined using Particle Size Analyzer (PSA) and Fourier-

Transform Infrared Spectroscopy (FTIR). PSA results showed an average particle size of 13.604 µm, classifying the material as micro-scale biochar (< 100 µm), which typically offers a higher surface area and better contact with adsorbates in aqueous systems, thereby improving pollutant removal efficiency [28]. FTIR analysis revealed nineteen distinct absorption bands between 459.56 cm^{-1} and 3866.01 cm^{-1} corresponding to functional groups such as hydroxyl (–OH), carbonyl (C=O), aromatic (C=C), ether (C–O–C), and sulfonate (S=O, S–O). Peaks in the 3866–3311 cm^{-1} region indicated –OH stretching of alcohol, phenol, and carboxylic acid groups, while the band at 1548 cm^{-1} represented aromatic C=C vibrations, reflecting the partially preserved lignin-derived carbon framework that enhances structural stability [29,30,31]. The presence of S–O and S=O bands (1045–1235 cm^{-1}) confirmed successful surface sulfonation induced by H_2SO_4 activation, increasing surface acidity and promoting electrostatic interactions with cationic contaminants [32,33]. These functional groups collectively enhance adsorption through hydrogen bonding, ion exchange, and complexation mechanisms. Cr(VI) reduction and subsequent Cr(III) complexation occur via these oxygen and sulfur containing groups, confirming the material's potential for heavy-metal adsorption [34, 35]. Overall, the A3B3 biochar exhibited micro-sized particles, well-developed porosity, and chemically active surfaces suitable for sustainable environmental adsorption applications.

4. Conclusions

This study successfully evaluated how pyrolysis temperature and H_2SO_4 concentration affect the characteristics of activated micro biochar from sugarcane bagasse. The results showed that pyrolysis temperature had a significant influence on all measured parameters, while H_2SO_4 concentration affected most parameters except methylene blue adsorption. Increasing the pyrolysis temperature and H_2SO_4 concentration improved the activation performance by decreased moisture, volatile matter, and ash content, also by increasing fixed carbon, iodine number, and methylene blue adsorption up to their optimal levels. The best treatment, based on the Multiple Attribute Zeleny method, was obtained at 600°C with 5M H_2SO_4 (A3B3). The activated micro biochar produced under these conditions met three of the six SNI 06-3730-1995 requirements and showed desirable characteristics, including high iodine adsorption (721.55 mg/g), large surface area (795.73 m^2/g), micro-sized particles (13.604 µm), and functional groups that enhance adsorption performance. Overall, the findings confirm that the optimized pyrolysis and activation conditions successfully improved the properties of micro biochar, demonstrating potential for Cr(VI) removal due to its developed surface area, micro-scale particle size, and active functional groups.

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