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Effect of temperature and heating rate on date palm biochar characterization

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Abstract: The role of biochar in improving soil fertility and carbon sequestration is widely recognized. To maximize its benefits in soil applications, it is essential to fully understand its physicochemical properties, which are largely shaped by the choice of feedstock and the specific pyrolysis conditions. This study investigated the effect of pyrolysis temperature (400, 550, and 700 °C) and heating rate (13 and 130 °C/min) on the yield and properties of biochar derived from date palm residues. The results demonstrated a significant decrease in biochar yield with increasing temperature: from 45.2% at 400 °C and 13 °C/min to 31.9% at 700 °C and 130 °C/min. At a fixed temperature, increasing the heating rate from 13 to 130 °C/min reduced the yield by approximately 5–8%, confirming the inverse relationship between thermal severity and char formation. Statistically significant differences ($p < 0.05$) were observed among treatments. Moreover, higher pyrolysis temperatures led to an increase in fixed carbon content (from 56.4% at 400 °C to 72.8% at 700 °C) and a reduction in volatile matter, indicating enhanced aromaticity and thermal stability. The surface area and porosity of the biochar also improved with temperature, suggesting better suitability for soil amendment, carbon sequestration, and pollutant adsorption. Overall, a pyrolysis temperature of 550 °C combined with a low heating rate (13 °C/min) provided an optimal balance between biochar yield (34.8%) and quality, supporting its potential use in sustainable agricultural and environmental applications.

Keywords: *Phoenix dactylifera* L; date palm residues; Biochar analysis ; slow pyrolysis ; Lignocellulose.

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1. Introduction

The significance of co-products such as biochar, derived from agricultural and forestry waste, particularly lignocellulosic biomass, has captured considerable attention within the scientific community. There is an increasing global initiative to develop strategies that transform agricultural waste into new value-added products instead of sending it to landfills [1].

The date palm tree (*Phoenix dactylifera* L.) is widely cultivated across the arid regions of the Arabian Peninsula, North Africa, and Iran, where it is integral to both local economies and diets. Algeria, as the fourth-largest global producer of dates, yields over 1.18 million tons of dates annually from more than 20 million date palms [2]. Each date palm tree generates about 30 to 50 kilograms of dry lignocellulosic waste per year, comprising fronds, leaves, and trunks, representing a largely untapped resource for agricultural enhancement [3,4]. Rather than being incinerated in open fields or disposed of in landfills, practices that carry significant environmental and health risks, this biomass can be repurposed into organic amendments like compost or biochar [5]. When properly processed,

date palm waste enriches the soil with organic matter, enhances moisture retention, and improves soil structure, making it particularly valuable for Algeria's arid regions, where soil fertility is limited [6]. This strategy addresses waste management challenges while promoting sustainable agricultural practices, supporting soil health, and reducing dependence on chemical fertilizers [7].

Biochar, in particular, stands out as an innovative, eco-friendly form of carbon. With its highly porous structure and inert characteristics, biochar has a low density and a high cation exchange capacity, making it exceptionally effective at improving soil health and fostering sustainable agriculture [8]. It is generated by the thermal decomposition of plant biomass in an environment with limited oxygen. [9]. Biochar's incorporation into soil has been shown to increase cation exchange capacity, nutrient retention, pH, and water-holding capacity, while reducing bulk density and enhancing crop yields [10]. Moreover, its high aromaticity confers significant resistance to biological and chemical degradation, rendering biochar an effective means of carbon sequestration The microporous structure of biochar enhances its ability to adsorb various organic and inorganic contaminants, making it valuable for applications in soil and water remediation. [11,12].

The quality of biochar is principally determined by pyrolysis parameters such as temperature, heating rate, biomass type, and particle size [13–15]. Pyrolysis temperature and heating rate significantly influence the formation and volatilization of intermediate compounds, thereby affecting biochar's physical and chemical attributes, including volatile matter, fixed carbon content, ash content, carbon percentage, and heating value [13]. Surface features of biochar, which impact its reactivity and combustion properties, are also critical considerations [16]. Polycyclic aromatic hydrocarbons (PAHs) and heavy metals (HMs) concentrations are significantly influenced by pyrolysis conditions. Higher pyrolysis temperatures generally lead to an increase in PAH content, as the thermal degradation of organic compounds accelerates, which also impacts the stability and mobility of HMs within biochar. This can pose serious environmental and health risks due to the potential leaching of these compounds into surrounding ecosystems [17,18].

Therefore, this research investigates the influence of pyrolysis temperature and heating rate on the physicochemical properties of biochar derived from date palm lignocellulosic waste. The study aims to assess the biochar's suitability for agronomic or environmental management, contributing to enhanced sustainability practices.

2. Materials and Methods

2.1. Biochar Production

The pyrolysis experiments were carried out in a muffle furnace, utilizing a stainless steel reactor with internal dimensions of 20.5 cm in length, 12.2 cm in width, and 7.5 cm in height. The sawdust of lignocellulosic waste (leaf sheath, leaves and pedicels) from date palm, with particle sizes ranging from 1 to 5 mm in diameter was placed inside the reactor, which was then securely sealed to maintain controlled conditions. Pyrolysis was performed for three hours at two distinct heating rates: a rapid rate of 130°C min^{-1} and a slow rate of 13°C min^{-1}, under limited oxygen conditions to ensure proper thermal decomposition. After pyrolysis, the reactor was allowed to cool gradually to room temperature within the furnace. The biochar samples were designated based on the heating rate and final temperature: 400, 550 and 700°C, and coded as flow; BCF400, BCF550, and BCF700 for biochars produced at the flow heating rate at 400, 550 and 700°C, respectively, and BCS400, BCS550, and BCS700 for biochars produced at the slow heating rate at 400, 550 and 700°C, respectively.

For subsequent analysis and potential applications, the biochars were first weighed and then ground to pass through a 0.3 mm (60 mesh) sieve. The biochar yield was determined using the following formula:

$$\text{Yield } \% = \frac{mass\ \ of\ \ biochar\ \ (g)}{oven\ \ dry\ \ mass\ \ of\ \ feedstock\ \ (g)} \times 100\%$$

2.2. Biochar characterization

2.2.1. Proximate analysis, pH, and conductivity measurements

Volatile matter (VM) and ash content were analyzed according to the American Society for Testing and Materials D5142 method [19]. VM content was determined by measuring the weight loss after heating the char to 950°C for 7 minutes. Ash content was assessed by measuring the weight loss after combustion at 750°C for 6 hours, without a ceramic cap. The fixed carbon content was then calculated using the following equation:

$$\text{Fixed carbon } \% = 100 \ \% - (Ash \ \% + Volatile \ matter \ \%)$$

pH and electrical conductivity (EC) were measured following a 30 minute shaking period at a 1:5 solid to water ratio, using a pH meter and a conductivity meter, respectively.

2.2.2. Biochar mineral elements analysis

The total concentrations of sodium (Na), potassium (K), and calcium (Ca) were determined using a flame emission spectrophotometer (Model PFP7, Jenway, UK) as reported by Jones (2001) [20]. For sample preparation, 0.5 g of oven-dried and finely ground plant material was digested using a wet acid digestion method. A mixture of 10 mL of concentrated nitric acid (HNO_3, 65%) and 2 mL of perchloric acid ($HClO_4$, 70%) was added to each sample in a digestion tube and heated at 180 °C for approximately 2 hours on a digestion block until a clear solution was obtained. After cooling to room temperature, the digests were filtered through Whatman No. 42 filter paper and the filtrates were diluted to a final volume of 50 mL using deionized water.

2.3. Statistical Analysis of Data

Each parameter was tested with at least three replications. A two-way ANOVA was conducted to assess the interaction effects between pyrolysis temperature and heating rate. Additionally, a one-way ANOVA was performed, followed by mean comparisons using a post hoc Tukey test, to evaluate differences between groups.

3. Results and discussion

3.1. Biochar yield

The physical characteristics of biochars derived from date palm waste are presented in Figure 1 and Table 1. Biochar yields were significantly affected by pyrolysis temperature, heating rate, and the interaction between these variables ($p<0.001$). Specifically, yields decreased as pyrolysis temperature and heating rate increased. Similar trends have been documented in the literature for various biomass sources [21,22]. The higher biochar yields observed at lower temperatures suggest that only partial pyrolysis of the feedstock material occurred, as reported by Angin (2013) [16] and Katyal (2023) [23].

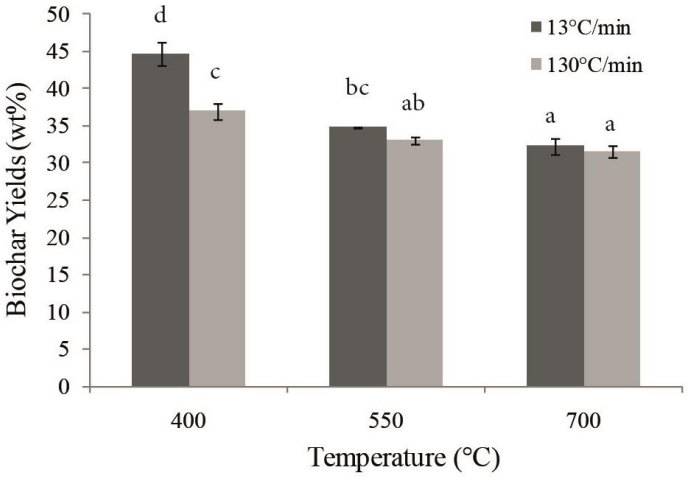


Figure 1. Effect of pyrolysis temperature and pyrolysis heating rate on date palm wastes biochar yields.

Table 1. Characteristics of date palm waste biochars produced at different pyrolysis temperatures and heating rates. (abbreviations: **pH**: *Potential of Hydrogen,* **EC**: *Electrical Conductivity)*

Pyrolyse Temperature °C	Heating rate (C min^{-1})	Ash %	Volatile matter %	Fixed carbon %	pH	EC
400	13	17±0a	30.7±1.2d	52.3±1.2b	9±0.2a	10.1±0.2c
550	13	22.3±0.6bcd	14±1b	63.7±1.5cd	9.1±0.3ab	14.0±0.6d
700	13	22±1bc	9±2a	69±1.7d	9.9±0.1c	14.6±0.4d
400	130	20 ±0ab	35.3±1.5e	44.7±1.5a	9.1±0.6ab	5.6±0.4a
550	130	25.7±2.5d	23±2.6c	51.3±5b	9.4±0.2abc	7.9±0.7b
700	130	24.3±1.2cd	15±1b	60.7±0.6c	9.8±0.4bc	13.4±1d
P values		0.000	0.000	0.000	0.002	0.000

The analysis of the effects of heating rate and pyrolysis temperature on biochar mass yields revealed that heating rate had a more pronounced impact at lower temperatures. At higher temperatures, the difference in yield between heating rates diminished. For instance, at 400°C, increasing the heating rate from 13°C/min to 130°C/min resulted in biochar yields ranging from 44.7% to 36.9%, a difference of 7.75%. Conversely, at 550°C and 700°C, the differences in yield were significantly reduced to 1.8% and 0.7%, respectively.

These results align with previous research findings [16,24,25]. For example, Parihar et al [25] demonstrated that biochar yields decline with increasing pyrolysis temperature, largely due to the enhanced primary and secondary decomposition of char residues. This suggests that temperature is a critical factor influencing biochar yield and composition, with practical implications for optimizing biochar production conditions.

3.2. Proximate Analysis

The interaction between pyrolysis temperature and heating rate did not exhibit a significant effect on ash content, volatile matter, or fixed carbon, while the influence of the single factor was highly significant (Table 1; $p < 0.001$). The non volatile and non combustible components of biochar are largely determined by its ash content. The ash concentration increased sharply as the pyrolysis temperature rose from 400°C to 550°C, ranging from 17.3% to 22.3% for runs at 13°C min^{-1} and from 20% to 25.7% for runs at 130°C min^{-1}. However, between 550°C and 700°C, the ash content remained relatively stable for both heating rates. Moreover, the ash content significantly increased with rising heating rates.

For various types of biomass, such as safflower seed press cake, olive mill residue, and orange peels, these ash values were higher than those reported by several researchers [16,22,26]. Nevertheless, they were consistent with values obtained for date palm biochar [13]. The high ash content in date palm biochar may result from interactions between organic and inorganic components during pyrolysis. Date palm trees, known for their high silicon content [27], facilitate the formation of Si-C bonds, which elevate pyrolysis temperatures and increase aromatic component concentration and biochar recalcitrance [28].

Table 2. Effect of pyrolysis temperatures and heating rates on mineral element of date palm waste biochars.

Temperature °C	Heating rate (C min^{-1})	Na (g kg^{-1})	K (g kg^{-1})	Ca (g kg^{-1})
400	13	2.54±0.04a	53.00±0.52c	11.33±0.58a
550	13	2.84±0.03a	62.01±0.26d	25.00±1.73c
700	13	3.61±0.08b	70.27±0.90e	15.33±0.58b

400	130	1,84±0.05c	34.53±0.69a	16.67±0.58b
550	130	1.96±0.04c	41.74±1.13b	27±1. 8c
700	130	2.96±0.03d	64.26±1.13d	14.33±1.15b
Anova		0.000	0.000	0.000

The concentration of volatile matter in the biochars decreased significantly as the pyrolysis temperature increased from 400°C to 700°C, dropping from 35.3% to 15.1% at a heating rate of 130°C min^{-1} and from 30.7% to 9% at a heating rate of 13°C min^{-1}. Additionally, volatile matter content declined sharply with rising heating rates (Table 1; $p < 0.001$). Similar findings have been documented for date palm and other feedstocks [8,9,13,16]. The low volatile matter content is often considered a predictor of biochar stability, with lower content suggesting higher stability [13,29].

Conversely, the fixed carbon content increased significantly as the pyrolysis temperature rose from 400°C to 700°C, moving from 52.3% to 69% at a heating rate of 13°C min^{-1} and from 44.7% to 60.7% at a heating rate of 130°C min^{-1}. These results align with the findings for date palm biochar [13]. The fixed carbon content also increased markedly with higher heating rates (Table 1; $p < 0.001$). The increase in fixed carbon percentage with temperature suggests a greater potential for recalcitrant carbon. Higher concentrations of polyaromatic carbon in biochar have been associated with increased biochar stability [30,31].

3.3. Biochar pH and EC

The pyrolysis temperature had a significant effect on the pH values of the biochar, while the interaction between temperature and heating rate did not have a notable impact on pH (Table 03). All biochar samples exhibited alkaline pH values, which increased significantly from 9 at 400°C to 9.9 at 700°C. Mukherjee et al. [32] explained that the increase in pH could be attributed to the release of alkali salts from organic compounds and the reduction of acidic functional groups.

Electrical conductivity (EC) was significantly affected by pyrolysis temperature, heating rate, and their interaction (Table 01; $p < 0.001$). The EC values showed an inverse relationship with both parameters: EC increased with temperature but decreased with heating rate. These findings are consistent with previous studies on various feedstocks [1,13,17,22,33]. Additionally, at a lower heating rate (13°C min^{-1}), EC showed a pronounced decrease at lower temperatures. Specifically, at 400°C, EC dropped from 10.1 to 5.6, and at 550°C, it decreased from 14 to 7.9. However, at higher temperatures (700°C), variations in EC due to heating rate were minimal.

Table 3. interaction effect of pyrolysis temperatures and heating rates on date palm waste biochars parameters. (abbreviations: **pH**: *Potential of Hydrogen,* **EC**: *Electrical Conductivity)*

	Ash	Volatile matter	Fixed carbon %	Yield %	pH	EC	Na	K	Ca
Temperature	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000	0.000
Heating rate	0.000	0.000	0.000	0.000	0.491	0.000	0.000	0.000	0.013
Temperature x Heating rate	0.776	0.111	0.232	0.000	0.501	0.000	0.004	0.000	0.000

The increase in EC can be attributed to the loss of volatile compounds and the enrichment of the mineral fraction, especially potassium (K^+) and sodium (Na^+) ions, in the ash [17,33,34]. This study found a strong correlation ($p < 0.01$) between EC and volatile matter (0.891), potassium (0.977), and sodium (0.916).

3.4. Biochar mineral elements

The concentrations of sodium (Na) and potassium (K) displayed a marked increase with both higher pyrolysis temperatures and heating rates. This behavior is consistent with the findings of Zhang et al. [15], who highlighted that elevated temperatures can enhance the release and concentration of these alkali metals in biochar. The increase can be attributed to the thermal breakdown of organic matter, which liberates these minerals and incorporates them into the ash fraction. As temperature rises, the release and redistribution of alkali metals become more pronounced, contributing to the higher concentrations observed.

On the other hand, the behavior of calcium (Ca) presented a distinct pattern. Unlike sodium and potassium, calcium concentration initially increased from 400°C to 550°C but then declined at 700°C. This trend suggests that calcium may undergo different chemical transformations during pyrolysis. The initial increase in calcium could be linked to the breakdown of calcium containing organic compounds and their subsequent integration into the biochar matrix. However, the subsequent decrease at 700°C could be explained by the volatilization of calcium compounds at high temperatures, a phenomenon also observed by Sun et al. [35] when studying hickory wood. The decrease in calcium concentration at elevated temperatures could be due to the volatilization and potential loss of calcium as part of gaseous or particulate emissions, especially as the stability of some metal oxides can diminish under extreme thermal conditions.

Pituello et al [36] further support this explanation, noting that the volatilization of metals, including calcium, becomes significant at high pyrolysis temperatures. This volatilization may lead to a reduction in the concentration of certain metals within the biochar, influencing its chemical composition and overall properties. Consequently, understanding these transformations is crucial, as they affect the biochar's nutrient availability and its potential applications in agriculture and soil amendment, where mineral content plays a critical role.

4. Conclusion

The pyrolysis temperature and heating rate significantly affected the characteristics of biochar generated from date palm. Elevated temperature augmented fixed carbon, ash, pH, electrical conductivity, and inorganic minerals (K, Na, Ca), while diminishing the yield and volatile matter of biochar. In comparison to pyrolysis temperature, an increased heating rate elevated the fixed carbon, ash, volatile matter, pH, and calcium content, while diminishing the yield, sodium, potassium, and electrical conductivity of biochar. Further research is necessary to determine the appropriate applications of date palm biochar characteristics for successful utilization in various purposes.

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