Advances in Science and Sustainability, 2026, 3–7

doi: 10.70728/susta.v02.i04.001
Volume 02, Issue 04
ISSN: 3060-5407
Paper

PAPER

STUDY OF THE ELEMENTAL COMPOSITION AND PHYSICOCHEMICAL PROPERTIES OF BIOCHAR OBTAINED FROM COTTON STALK WASTE BY PYROLYSIS

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Abstract

In this study, the authors developed a thermochemical loading method for producing biochar through the pyrolysis of cotton waste, and conducted a physicochemical analysis of the results. The cotton waste was thermally processed at a temperature range of 500÷600 °C to obtain high-carbon, solid heat biochar. Using the PQ-100 "Pipe-in-a-Pipe" type pyrolysis device (Figure 1), high-carbon, porous, multifunctional biochar samples were obtained from the cotton waste, the subject of experimental research (Figure 2). The elemental composition of the biochar samples was analysed using energy-dispersive X-ray spectroscopy (EDS), and the amounts of the available products were determined: carbon (70÷72%), oxygen (11÷15%), hydrogen (3.0÷3.1%), nitrogen (0.8÷1.8%) and nitrogen (0.8÷1.8%). EDS spectrum analysis indicates that the biochar has a high carbon content.

Key words: Cotton waste, biochar, pyrolysis methods, pyrolysis device, elemental composition, X-ray spectroscopy.

Introduction

"Biochar" is a term commonly used to describe a solid product formed by heating carbonaceous materials at high temperatures in an airless environment. According to analytical data, biochar products have a high carbon content. Coal fuel is a clear example of this, and is the oldest known fuel used for producing fire or thermal energy. Biochar is obtained from local biomass and agricultural waste using various methods, providing an alternative to coal. It is made from organic compounds, particularly forest and agricultural waste products, and has a very low oxygen content

Compiled on: March 2, 2026.

compared to ordinary coal. Thus, biochar is formed by the thermal processing of biomass in an oxygen-free environment at relatively low temperatures (below 700°C) [1]. Biochar can be produced from agricultural waste for various purposes: firstly, to improve soil quality by storing carbon and increasing nutrient availability, reducing density, improving pH levels and increasing porosity and moisture retention; secondly, to filter water as an adsorbent; thirdly, to insulate in the construction industry; and fourthly, to accelerate methane production during anaerobic digestion by adding it to animal excrement and other raw materials without percolation. The properties of the raw carbonized organic matter and the operating modes used in the production process are determined by the biochar production process. The main methods of biochar production are pyrolysis (either slow or fast) and gasification [2,3]. The physicochemical properties of the resulting biochar depend directly on the type of biomass raw material used, its chemical composition, the method of production and the production technology. Most organic substances begin to thermochemically decompose at temperatures above 120 °C.

Hemicelluloses undergo thermochemical decomposition at temperatures between 200 and 260 °C, cellulose at temperatures between 240 and 350 °C, and lignin at temperatures between 280 and 500 °C [4]. Therefore, the ratio of these components determines the degree of reactivity and, consequently, the extent of change in the physical structure during processing. Biochar is characterised by high porosity, with pores ranging in size from micro to macro. The large pores formed by the vascular bundles of the raw biomass material are important for improving soil quality as they provide a habitat for symbiotic microorganisms. The increasing demand for charcoal in various fields is leading to an expansion in the production of biochar. One of the most widely used modern technologies for producing biochar is the thermochemical transformation process. This process includes pyrolysis, hydrothermal carbonisation, gasification, torrefaction and hydrothermal liquefaction [5-9].

Methods and materials

Biochar was obtained from cotton waste using the pyrolysis method. This involved pre-drying the waste and then processing it thermally at 500÷600 °C in an oxygen-free environment. The elemental composition of the biochar samples obtained was determined using energy-dispersive X-ray spectroscopy (EDS) in conjunction with a scanning electron microscope. Based on the EDS analysis, the presence of the following components in the biochar was determined: carbon, hydrogen, oxygen, nitrogen, sulphur and mineral residue. The relative mass fraction of each element was also estimated.

Pyrolysis is a thermochemical process used to produce biochar, biofuel (bio-oil) and synthesis gas (pyrolysis gas) from biomass feedstock [9-12]. The process involves heating the feedstock in an oxygen-free environment at temperatures ranging from 400°C to 1200°C, followed by thermochemical decomposition under the influence of heat. This allows the feedstock to be heated above its thermal stability limit, resulting in more stable products containing solid residues, such as biochar. Additionally, creating an oxygen-free environment during pyrolysis ensures that the biomass does not burn when heated. Pyrolysis is a very complex process involving various reactions in the reaction zone [10]. Another study reported that the low temperature range of pyrolysis is from 250°C to 900°C. Agricultural waste is composed of lignin, cellulose, hemicellulose and silica. Cellulose pyrolysis is typically carried out at 350÷380 °C and lignin pyrolysis at 300÷500 °C [8]. The efficiency of the biochar production process depends on different operating conditions; however, the efficiency of biochar production is usually maintained at low temperatures for a long time to achieve high efficiency and quality [9]. Therefore, the temperature range should be 300÷700°C to ensure the efficient pyrolysis of biomass feedstock. Heavy chemical compounds are decomposed by secondary or fast pyrolysis, converting the biomass into biochar or alternative liquid and gaseous fuels.

Varying the pyrolysis temperature used to produce biochar can determine its utilisation effect for both single feedstock and biochar production. A study by Bardi et al. reported that biochars produced at pyrolysis temperatures ranging from 350 to 900 °C had a positive effect on anaerobic digestion efficiency (from different feedstocks). Therefore, further studies are needed to investigate how the properties of biochars obtained from specific feedstocks change with pyrolysis temperature and the effect this has on anaerobic digestion.

Pyrolysis temperature plays an important role in determining the physicochemical and physicothermal properties of biochar. Typically, biomass feedstocks are freed from moisture and volatiles at temperatures up to 200 °C, hemicellulose and cellulose decompose and volatilise at temperatures up to 500 °C, and strongly bound organic matter (such as lignin) degrades at temperatures above 500 °C (24). Biochar porosity and specific surface area generally increase with increasing pyrolysis temperature due to the decomposition of the biomass feedstock and the formation of micropores (25).

However, temperatures above 700 °C can lead to structural ordering and pore coalescence, resulting in a reduction in surface area (26). Biochars produced at higher temperatures have higher pH, carbon and ash content [10]. Biochar has been produced from agricultural waste at temperatures of 450, 600, 750 and 900 °C. The agricultural waste was cut into cubes and cylinders measuring 5÷10 mm and heated at a temperature ranging from 80 to 100 °C for 24 hours to obtain the biochar. The dried raw material was manually placed in the pyrolysis reactor and then purged with nitrogen. For each production temperature, the reactor was heated at a rate of 10 °C/min and held at the target temperature for 1.5 hours. The resulting biochar had a size ranging from 4 to 8 mm.

Results and discussions

Below is a pilot version of the experimental copy of the PQ-100 "Pipe-in-a-Pipe" pyrolysis device.

Figure 1. Experimental copy of the PQ-100 "Pipe-in-a-Pipe" pyrolysis device.

Figure 2 shows a sample of biochar obtained from the thermal processing of cow dung waste by pyrolysis at 500–600 °C during the research conducted. Table 1 presents its elemental composition.

Table 1.

Elemental composition of biochar obtained from the pyrolysis of cotton waste

No	Item name	Marking	Amount, % (by mass)
1	Carbon	C	70–72
2	Hydrogen	H	3.1–3.0
3	Oxygen	O	11–15
4	Nitrogen	N	0.8–1.8
5	Sulfur	S	0.1–0.2
6	Ash content	A	15–8

We determined the elemental composition of biochar samples obtained by pyrolysis from cotton waste using energy-dispersive X-ray spectroscopy (EDS). This analysis was carried out using an EDS detector equipped with a scanning electron microscope (SEM). The results are presented in Figure 3.

Таблица 4

Матричная форма калькуляции затрат на производство и реализацию продукции, в условных единицах[44]

Наименование затрат (расходов)	**Производ. себест.**	**Расходы периода**	**Затраты, всего**
1.Текущие материальные и приравненные к ним затраты **(ТМПЗ),** всего в том числе	55,0	15,0	70,0
1.1.Материальные затраты	33,0	2,0	35,0
1.2.Услуги сторонних организаций	17,0	3,0	20,0
1.3.Рентные платежи за использование человеческих (взносы на социальное страхование) и природных ресурсов (включая налог на недра)	0	8,0	8,0
1.4.Экологические платежи и компенсационные выплаты	5,0	2,0	7,0
2.Добавленные затраты **(ДЗ),** всего в том числе:	20,0	10,0	30,0
2.1. Амортизация (текущий и кап. ремонт основных средств)	15,0	5,0	20,0
2.2. Фонд оплаты труда (ФОТ), с учётом НДФЛ, без социальных страховых начислений	5,0	5,0	10,0
3.Затраты (п.1+п.2), всего	75,0	25,0	100,0

Figure 3. EDS elemental spectrum analysis of biochar from the pyrolysis of cotton waste.

The biochar sample obtained by pyrolysis from the gasification plant waste was first dried in a muffle furnace, after which the biochar was ground into a fine powder to prevent agglomeration. The samples were then placed on the SEM stand using double-sided conductive carbon tape. To improve electrical conductivity, a thin layer of gold (Au) or carbon was coated onto the sample surface. When an electron beam was then directed onto the biochar sample surface, electrons were emitted from the inner shells of the atoms and X-rays with specific energies were generated as they transitioned to the vacated energy levels. These X-rays were recorded using an EDS detector as a spectrum in energy (keV)-intensity (a.u.) coordinates. Each peak was identified according to the characteristic radiation line ($K\alpha$ or $K\beta$) of the corresponding element. Based on EDS analysis, biochar is mainly composed of carbon (C), hydrogen (H), oxygen (O), nitrogen (N) and sulphur (S), as well as mineral components. The ash content is summarised as A. The relative mass fraction of the elements was calculated using a semi-quantitative EDS program. EDS spectrum analysis of biochar obtained from the thermal processing of cotton waste by pyrolysis showed that it contains a high mass fraction of carbon (C).

Conclusions

The high carbon content indicates that organic matter decomposes effectively during the pyrolysis process, forming a high-carbon structure. In general, EDS analysis shows that biochar is composed of a high proportion of carbon and relatively small amounts of various organic compounds and mineral residues. This indicates its potential use as a solid fuel for preparing water-coal alternative fuel, an adsorbent, and a soil improver.

EDS analysis can also be used to assess the morphological structure and mineral composition of biochar, as well as to determine the efficiency of the pyrolysis process.

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