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Changes of C, H, and N Elements of Corn Straw during the Microwave Heating Process

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Abstract: Due to the rapid growth of the global economy, energy consumption has been steadily increasing, leading to increasing issues such as energy shortages and environmental concerns. Biomass energy, a critical renewable energy source, plays a vital role in advancing low-carbon energy development and resource sustainability. In this study, experiments were conducted to study the migration of C, H, and N elements of corn straw during the microwave heating process, and the effects of residence time, heating temperature, and microwave power were also investigated. The results showed that when the temperature rose, both the proportion of C and H elements fluctuated slightly. Specifically, when the temperature rose from 75 °C to 275 °C, there was a 1.02% increase in the proportion of the C element and a 0.25% decrease in the proportion of the H element. Residence time appeared to be a significant factor influencing the changes in C, H, and N elements. For a 40 min residence time, the proportion of the C element increased from 31.77% to 35.36%, while the proportion of the H element decreased from 4.50% to 3.83%. When there was an increase in the microwave power between 160 W and 200 W, higher temperatures were reached in the samples, leading to the carbonization process of corn straw being more complete. Consequently, the proportion of the C element rose with extended residence time, whereas the proportion of the H element decreased as the residence time increased.

Keywords: C; H; N; corn straw; microwave heating

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1. Introduction

With the rapid advancement of the global economy and the steady rise in population, energy consumption is increasing year by year [1,2]. The excessive exploitation and consumption of fossil fuels have led to a growing list of drawbacks. Energy shortages and environmental crises have gradually emerged as the primary challenges confronting the international community. The issue of climate change resulting from excessive carbon dioxide emissions is widely recognized as the most severe environmental problem of our time, and it represents a crucial strategic concern for global sustainable development. Notably, more than 90% of carbon dioxide emissions linked to energy production originate from the burning of fossil fuels. Consequently, reducing the consumption of fossil energy is a necessary step to mitigate carbon emissions [3,4]. Renewable energy, characterized by abundant reserves and minimal environmental impact, is viewed as an effective alternative to fossil fuels and has garnered significant global attention [5].

Biomass energy, a key component of renewable energy, boasts remarkable qualities such as renewability, widespread availability, and low carbon emissions. It holds substantial potential for future development [6]. China, being a major agricultural nation with a dense population, possesses abundant biomass resources. In 2022, China's biomass production reached 3.7 billion tons, equivalent to 460 million tons of standard coal. However, the country's energy utilization rate of biomass is merely 12%, and its contribution to the overall energy consumption structure stands at only 4.6%, considerably lower than the

global average [7]. Therefore, actively utilizing and exploiting biomass energy resources holds immense significance in promoting low-carbon energy development and fostering resource recycling.

Presently, China's utilization of biomass energy exhibits a diversified trend, encompassing various approaches such as direct combustion, biogas fermentation, biomass gasification, and biomass oil production [8,9]. Notably, the products derived from biomass gasification and biomass oil production can effectively serve as substitutes for fossil fuels like oil and natural gas and have thus experienced substantial growth in recent years.

Microwave heating represents a novel approach to heating. In contrast to traditional heating methods, microwave heating offers distinct advantages, including even heating, rapid heating rates, high efficiency, and minimal environmental impact. These attributes have gathered significant attention from researchers [10–12]. Moreover, microwave-assisted chemical conversion technology has emerged as a central pathway for the utilization of biomass energy.

Qiu et al. [13] conducted a study on the impact of electric pyrolysis versus microwave pyrolysis of corn straw on biochar yield. The findings indicated that when subjected to microwave pyrolysis at 800 °C for 180 min, the biochar yield increased by 21.24% compared to electric pyrolysis. Shvets et al. [14] investigated the influence of microwave power on the product composition during the microwave pyrolysis of woody biomass. Their research revealed that as microwave power increased from 840 W to 1760 W, the average yields of CO and CO_2 remained relatively stable. However, with a further increase in power to 2200 W, there was a notable increase in the average yields of CO and CO_2. Additionally, high microwave power was found to enhance the production of CH_4 and H_2. Zhang et al. [15] developed a microwave-assisted biomass air gasification system. They subsequently investigated the effects of the airflow rate, gasification temperature, and residence time on the yield of syngas and carbon conversion rate. The outcomes demonstrated that increasing the residence time significantly elevated both syngas yield and carbon conversion rate. Specifically, under conditions of an airflow rate of 600 mL/min and a temperature of 700 °C, the residence time played a critical role in the process: at 30 min, the syngas yield can reach 76.14%. Fu et al. [16] carried out a study on microwave-assisted biomass air gasification in a fluidized bed reactor and obtained the optimal reaction conditions for syngas production: a gasification temperature of 900 °C, equivalent ratio of 0.35, and syngas yield of 78.2%. Mao et al. [17] carried out microwave-assisted pyrolysis experiments of furfural residue in a continuous spiral reactor and explored the effects of kaolin, CaO, and K_2CO_3 additives on the yield of pyrolysis gas. The results showed that all three additives can increase the yield of H_2 and CH_4, and CaO can also reduce the yield of CO_2.

The microwave absorption capacity of biomass is weak. In order to meet the temperature conditions of biomass gasification and pyrolysis, it is often necessary to add microwave absorbers, such as SiC, activated carbon, graphite, etc. Shi et al. [18] used bamboo, cork, pine, and mahogany as raw materials to study the effect of activated carbon additives on the production of syngas under microwave irradiation and found that the coupling of activated carbon reforming and microwave-assisted pyrolysis not only increased the yield of gas products and reduced the yield of bio-oil, but also further improved the selectivity of H_2 generation and increased the overall low heat value of products. Zhou et al. [19] studied and developed a continuous microwave-assisted pyrolysis system with a large processing capacity. Using SiC as the microwave absorber, it could process 10 kg of biomass per hour. Huang et al. [20] used magnetite as a microwave absorber to pyrolyze chlorella and spirulina to produce bio-oil. The results showed that Fe_3O_4 accelerated the decarboxylation reaction in the decomposition process of microalgae and promoted the formation of nitrogen-containing fatty compounds.

The bio-oil obtained by microwave pyrolysis of biomass has high oxygen content, low calorific value, complex composition, and instability and often needs to be upgraded by catalysis. Commonly used catalysts include metal salts, metal oxides, and ZSM-5 catalysts.

Li et al. [21] modified the ZSM-5 catalyst with NaOH and TPAOH alkaline solutions and applied it to microwave catalytic pyrolysis of rice husks. The results showed that the catalyst treated with 2.0 mol/L TPAOH solution showed better catalytic performance and the relative content of hydrocarbons in the pyrolysis products was high. The relative contents of benzene, toluene, and xylene were up to 58.6%. Wan et al. [22] applied $MgCl_2$ to the microwave catalytic pyrolysis of poplar wood, and the results showed that the furfural content in the pyrolysis products increased with the increase in MgCl.

Previous studies focused on the pyrolysis and gasification characteristics of biomass with the addition of microwave absorbents and catalysts but ignored the effect of microwaves on the heating characteristics of biomass. In this study, the effects of residence time, heating temperature, and microwave power on the migration of C, H, and N elements of corn straw were investigated by experiments without adding microwave absorbent, which is of great significance for the efficient utilization of biomass.

2. Materials and Methods

2.1. Materials

China is rich in straw resources, accounting for about 20% of the total biomass resources. In this study, samples of corn straw were taken from Lianyungang City, China, in 2022. Ultimate analysis was performed using an Organic Elemental Analyzer (Thermo Fisher Scientific Inc, Waltham, MA, USA), and the results are shown in Table 1.

Table 1. Proximate analysis and ultimate analysis results of corn straw.

Proximate Analysis (wt.%)				Ultimate Analysis (wt.%)				
M_{ar}	V_{daf}	FC_{ar}	A_{ar}	C_{ar}	H_{ar}	O_{ar} [a]	N_{ar}	S_{ar}
5.11	37.55	12.14	75.45	31.77	4.50	1.24	0.66	0.90

[a] Calculated by difference. M, V, FC, and A indicate moisture, volatile, fixed carbon and ash, respectively. C, H, O, N, and S indicate elements C, H, O, N, and S, respectively. Subscript ar means "as received basis". Subscript daf means "dry ash-free basis".

The corn straw was crushed in a Baihaojia HY-608 pulverizer (Hongtaiyang Electromechanical Inc., Yongkang, China), and then the crushed material was screened with an 80-mesh sieve; the corn straw below the sieve was taken as the experimental sample, as shown in Figure 1. In order to ensure that the composition of the sample does not change, contact with air should be avoided as much as possible, and it should be stored in a dry and airtight container before use.

Figure 1. Corn straw used.

2.2. Experimental System

The microwave heating device used in this study is shown in Figure 2 and mainly consists of two systems: (a) a microwave heating system; and (b) a temperature measurement system. Microwave heating systems include quartz tubes and microwave ovens. The

microwave oven is made by Beijing Yihuida Microwave Devices Co., LTD. (Beijing, China), with a length of 30 cm, a width of 30 cm, and a height of 35 cm. In the quartz tube setup, a small sieve plate is positioned in the middle section. This serves a dual purpose: it facilitates the loading of experimental samples and ensures that both the upper and lower layers of the sample are exposed to the surrounding air. To monitor temperature, a temperature measurement system composed of a digital thermometer and a K-type thermocouple is employed. The digital thermometer is responsible for gathering temperature data from the thermocouple probe and transmitting it to the computer through a USB connection. Once the arrangement of the quartz tube is finalized, the setup is insulated by adding thermal insulation cotton inside the furnace and wrapping the quartz tube. This precaution is taken to prevent any interference from radiation heat transfer, which could affect the accuracy of experimental results.

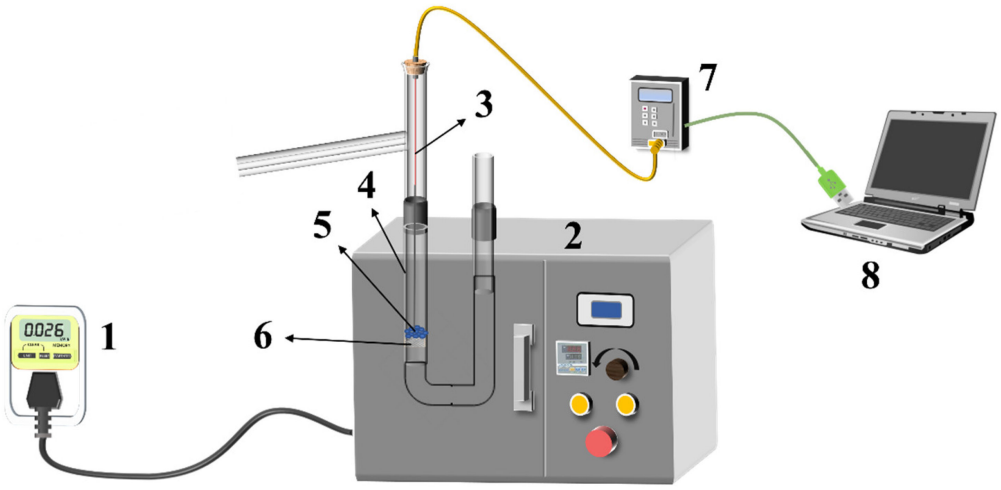


Figure 2. Microwave heating system. (1) power supply; (2) microwave; (3) K-type thermocouple; (4) reactor; (5) samples; (6) sieve plate; (7) digital thermometer; (8) computer.

2.3. Experimental Procedures

Connect the experimental device as shown in Figure 2 and open the temperature recorder to record the temperature data in real time. It should be emphasized that the diffusion rate of air in the sample was very slow without external force. Therefore, a small amount of sample was used in each experiment to ensure that the thermocouple temperature probe was just submerged by the sample.

Start heating by activating the microwave oven. Adjust the microwave power to reach the desired experimental temperature while maintaining a consistent temperature range or slight fluctuations within ±15 °C. This step aims to investigate the impact of temperature and residence time on the migration of C, H, and N elements. The effect of microwave power on the migration of C, H, and N elements was investigated by controlling the heating time. After the experiment was completed, the sample was cooled naturally and removed from the glass tube, and the change rule of C, H, and N elements was explored by ultimate analysis, which was performed using Thermo's Flash Smart Organic Element Analyzer.

To determine an appropriate experimental temperature, a preliminary experiment was conducted. Figure 3 shows the temperature of corn straw particles at 200 W. It was observed that the sample gradually heats up to 292 °C. Afterward, the temperature began to increase rapidly, reaching 1200 °C within 2 s. Simultaneously, a dense white smoke formed within the reactor. Evidently, at this point, a combustion reaction occurred within the sample rather than slow oxidation. To prevent a combustion reaction, the sample temperature must be maintained below 292 °C.

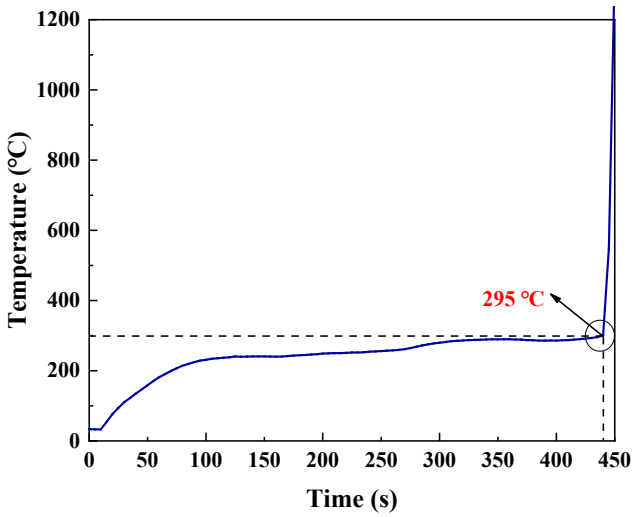


Figure 3. Temperatures of corn straw particles at 200 W.

3. Results and Discussion

3.1. Effect of Temperature on Migration of C, H, and N Elements

The reaction time was set at 10 min, and the proportions of C, H, and N elements changed at various temperatures were examined, as illustrated in Figure 4. When the temperature increased, the proportion of the N element remained nearly constant, with only a minimal 0.047% difference between the maximum and minimum values, which could be disregarded. This stability was due to the fact that N in biomass primarily existed in organic matter form, which was not easily vaporized at lower temperatures [23].

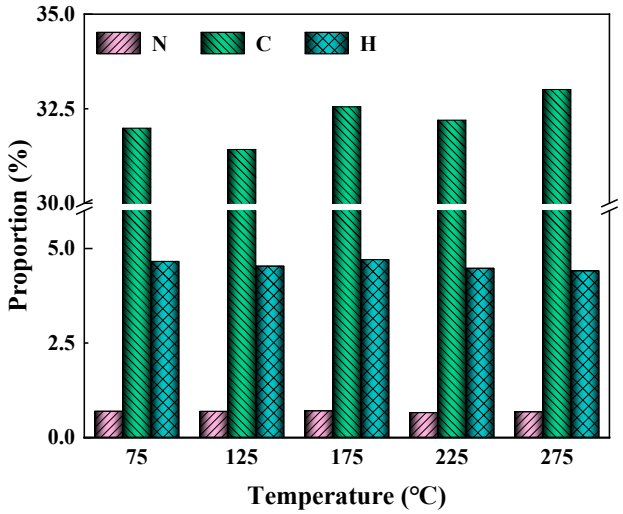


Figure 4. Proportions of C, H, and N elements at different temperatures (10 min).

Since the biomass contained a significant amount of water, it required lower temperatures for the water to vaporize. As the temperature rose, more water vaporized, causing the proportion of H element to steadily decrease. The proportion of the C element showed an overall upward trend. On the one hand, water and some volatiles were released, and the content of the H and O elements decreased, which in turn increased the proportion of the other elements. On the other hand, most C-containing components started to decompose at temperatures above 300 °C. Below 275 °C, the C element underwent only slow oxidation, resulting in minimal changes. For instance, at 275 °C, the proportion of the C element was 33.01%, while the proportion of the H element was 4.41%. In comparison to 75 °C, this

represented a 1.02% increase in the C element proportion and a 0.25% decrease in the H element proportion, as shown in Figure 4.

3.2. Effect of Residence Time on Migration of C, H, and N Elements

Temperature plays a pivotal role in the release of volatile compounds and the oxidation of element C. At lower temperatures, biomass samples primarily underwent water release, with minimal involvement in other reactions. Based on pre-experiment findings, a temperature of 275 °C was selected for further investigation into the impact of residence time on the migration of C, H, and N elements, as depicted in Figure 5. Over the initial 0–20 min, an increase in time led to a continuous rise in the proportion of the C element and a concurrent decline in the proportion of the H element. This phenomenon could be attributed to the volatilization of water and certain biomass components containing H. By the 30 min mark, there was a notable shift compared to the state at 20 min. The proportion of the C element decreased from 34.05% to 32.24%, and the proportion of the H element continued to decrease. This was due to the continuous release of volatile fractions containing H, further reducing the proportion of the H element. Meanwhile, the C element began undergoing a noticeable oxidation reaction, with a portion converting into CO_2 and being released. Comparing the color of the samples after the experiment, it can be found that when the residence time was 30 min, some samples became black. This resulted in a significant decrease in the proportion of the C element, overtaking the release rate of the H element. As time progressed to 40 min, the proportion of the H element continued to decline, with an accelerated rate, decreasing from 4.32% at 30 min to 3.83%. Conversely, the proportion of the C element increased notably, from 32.24% at 30 min to 35.36%. At this stage, the slow oxidation reaction of the C element had essentially concluded. The reduction in the proportions of H elements became the primary factor driving the increase in the proportion of the C element. These findings demonstrated that residence time is a crucial factor influencing the slow oxidation reaction of the C element, with the 30 min mark serving as a crucial point. The period between 20 and 30 min represented the active reaction time, while after 30 min, the reaction nearly ceased.

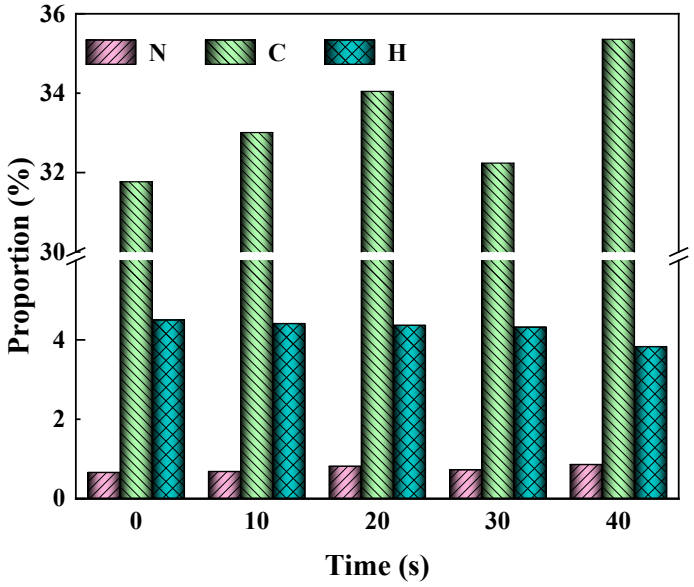


Figure 5. Proportion of C, H, and N elements at different times (275 °C).

3.3. Effect of Microwave Power on Migration of C, H, and N Elements

Based on the preliminary experiment, microwave power settings of 160 W, 180 W, and 200 W were selected. Figure 6 illustrates the temperature of corn straw particles during the initial 20 min at different microwave powers, with peak temperatures reaching 144 °C, 193 °C, and 233 °C, respectively. The temperature changes in corn straw followed a similar

pattern at other time intervals. At the 40 min mark, the maximum attainable temperatures for the corn straw were 165 °C, 223 °C, and 257 °C, respectively.

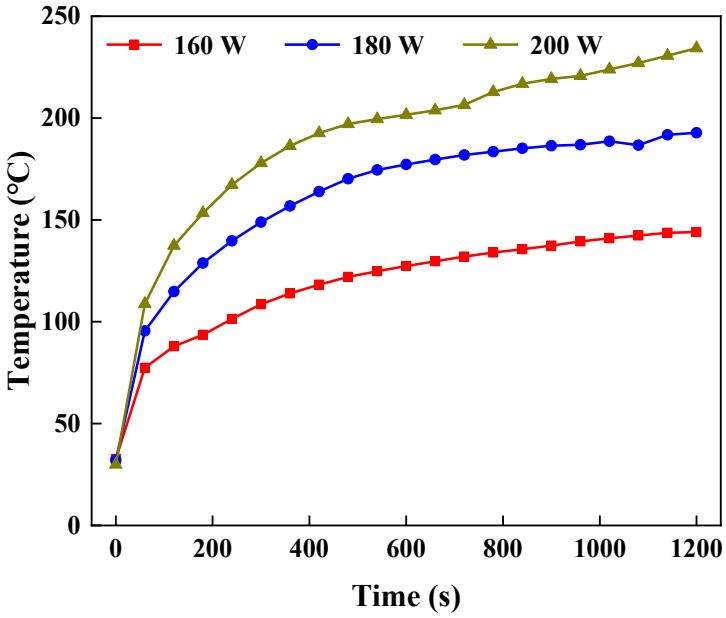
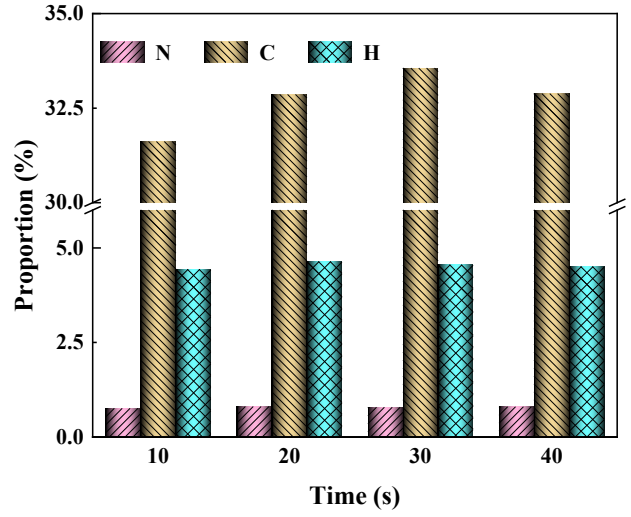


Figure 6. Temperatures of corn straw particles at different microwave powers.

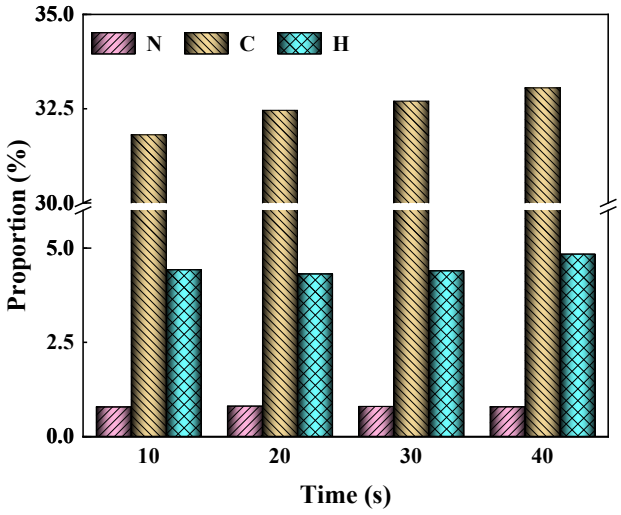
Figure 7 displays the changes in the proportion of C, H, and N elements at various microwave powers. Across the three microwave power settings, the changes in C, H, and N elements followed a consistent pattern: the proportion of the C element increased with time, the proportion of the H element decreased with time, and the proportion of the N element remained relatively stable with time. At the 10 min mark, the proportions of the C element for 160 W, 180 W, and 200 W microwave power were 31.62%, 31.81%, and 32.28%, respectively. During this time, the sample heating rates corresponding to these microwave power levels were as follows: 16.5 °C/min, 17.8 °C/min, and 24.4 °C/min. According to the relevant literature, the heating rate significantly affects the release of volatile gases [24], and the change in the proportion of the C element confirmed this observation. With an increased heating rate, the release of volatile gases containing H was accelerated, resulting in a decrease in the H element content and an increase in the C element proportion.

In the case of 200 W, the proportion of the H element changed very little over time. At 10 min, the H element content was 4.48%, and at 40 min, it was 4.42%, which was negligible. This indicated that most of the volatiles containing H releasable under this power condition have been released within a certain time. Consequently, even as time progressed, the volatiles containing H were no longer released, and the proportion of the H element remained almost constant until a higher temperature condition for further volatile release was reached. Different from the previous section, the microwave power was not adjusted during the experiment in this section, so the migration rule of C and H elements was different.

When the microwave power was 160 W and the time exceeded 30 min, the proportion of the C element began to decline, which was because the oxidation rate of the C element exceeded the release rate of volatile fractions containing H. At the same time, when the microwave power was 180 W and 200 W, the proportion of the C element continued to maintain an upward trend. It was proved that the temperature required for the oxidation of the C element was significantly lower than the temperature containing H volatile release. With the increase in microwave power, the maximum temperature that core straw can reach increased, and the release rate of volatiles containing H was accelerated. Although the oxidation rate of element C was also accelerated, the former rate was faster, so the proportion of element C will continue to rise.



(a)



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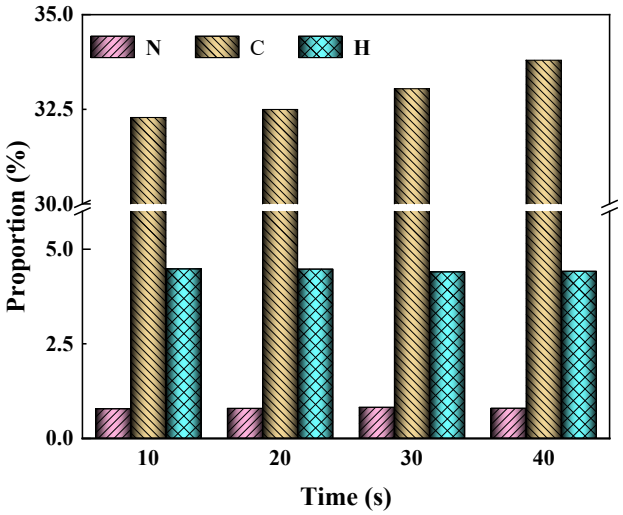

(c)

Figure 7. Proportions of C, H, and N elements at (**a**) 160 W, (**b**) 180 W, and (**c**) 200 W.

4. Conclusions

In this study, the effects of temperature, residence time, and microwave power on the migration of C, H, and N elements of corn straw during the microwave heating process were experimentally investigated. The following conclusions were obtained:

At temperatures below 275 °C, N elements of corn straw particles did not migrate much. As the temperature increased, the carbonization process of corn straw was more complete, and the proportion of the C element increased gradually. At 225 °C, the proportion of the C element decreased due to an oxidation reaction.

The residence time was critical for the migration of C, H, and N elements. The increase in residence time made the carbonization process of corn straw more complete, resulting in an increase in the C element proportion from 31.77% to 35.36%. A significant decrease in the proportion of the C element occurred around the 30 min mark, indicating a notable oxidation process.

Microwave power played a role in both the heating rate of the sample and the maximum temperature it could reach, which affected the changes in C, H, and N elements. At 160 W, the proportion of the C element increased initially and then decreased as residence time grew, with the turning point occurring at 30 min due to the oxidation of the C element. The proportion of the H element decreased as residence time increased due to the continuous release of H-containing volatiles. When the microwave power was 180 W and 200 W, the carbonization process of the C element was more complete with the increased residence time. Consequently, the proportion of the C element increased, while the proportion of the H element decreased.

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Data Availability Statement: Data are contained within the article.

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

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