Experimental Study on Compression Moulding of *Carya cathayensis* Husks at Room Temperature

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Abstract

Using crushed hickory husks as raw materials, a single factor test was carried out on a hydraulic-plunger forming machine with different moisture contents and length–diameter ratios (LDRs) of forming mould in order to obtain biomass moulding fuel (BMF) briquettes. The moulding pressure in the forming mould was measured by the sensor, and the microstructures of the BMF briquettes were observed via stereomicroscope. Results showed that hickory husks could be compressed and formed into BMF briquettes at room temperature. When the moisture content of the raw materials was 14 percent and LDR of the forming mould was 4.5, the quality of BMF was best, and relaxation density of the BMF briquette was 0.98 g/cm^3. When the moisture content stayed same, the forming pressure increased correspondingly with the increase of the LDR of the forming mould. When the LDR of the forming mould stayed the same, the moulding pressure increased first and then decreased with increase of moisture content. This study is of great significance because it demonstrates improvement in the utilization range of hickory husks, with the potential for reducing environmental pollution and increasing farmers' income.

Biomass energy has always been an important energy source for human survival, ranking fourth in the world's total energy consumption after coal, oil, and natural gas (Vassilev et al. 2015, Suh 2016). At present, the world is facing the crisis of fossil energy exhaustion. Biomass energy, as the only renewable carbon source currently used, is an internationally recognized zero-carbon-emission energy that is rich in potential sources, clean low-carbon, renewable, and has other characteristics (Man et al. 2017, Iftikhar et al. 2019, Dinesha et al. 2019). China is rich in biomass resources, and the annual available biomass resources amount is about 600 to 700 million tons of standard coal, of which 247 million tons of standard coal could be used for energy conversion, and this amount will further increase with the large-scale development of energy plants in the future (Zhang 2018). Biomass moulding fuel, abbreviated as "BMF," uses agricultural and forestry residues such as wood chips, sawdust, bamboo chips, straw, rice bran, bagasse, and fruit husks as raw materials through grinding, mixing, extrusion, and drying processes. They can be made in specific densities and shapes (such as pellet, briquette, block, rod, granular, etc.) and directly burned as a new type of clean fuel (Guan et al. 2020). BMF is currently the main form of biomass utilization, especially in the industrial field. Compared with biomass raw materials, BMF has higher density, higher calorific value, more convenient transportation, and 70 percent higher thermal efficiency (Saidur et al. 2011). BMF can increase the density of ordinary biomass from 40–150 kg/m^3 to 1,000–1,400 kg/m^3, not only saving transportation and storage costs but also greatly improving its combustion status. Thermal efficiency of direct burning of crop straw is only 10 to 30 percent, while thermal efficiency of BMF is up to 89 percent. In addition, emission of NOx of BMF is only one-fifth that of coal, and emission of SO_2 is only one-tenth that of coal, so BMF is an efficient and clean fuel (Suopajärvi

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et al. 2013, He et al. 2019). BMF has the advantages of carbon neutrality, low nitrogen and sulfur content, and less fluel gas pollution, etc. (Moron and Rybak 2015). In addition to biomass gasification, direct combustion, and mixed combustion power generation, it also can be widely used in heating boilers and other combustion equipment, which can significantly consume agricultural and forestry residues, reduce air pollution, and achieve zero CO_2 emissions, in line with the current concept of sustainable development of society. It is of great significance to the development of a low-carbon economy and establishment of a conservation-oriented society.

Currently, BMF production in Germany, Sweden, Finland, Denmark, Canada, and the United States can reach >20 million tons per year (Ma et al. 2019). However, development of BMF in China is still behind development in these countries. Although many researchers have studied BMF utilization methods and technology in China (Zhao 2016; Guan et al. 2017, 2020; Man et al. 2017; Xiang 2017; Wang et al. 2018; Lu 2016), there is no research on BMF production using hickory husks. Chinese hickory (*Carya cathayensis* Sarg.) is a well-known Chinese nut, widely distributed in the Zhejiang and Anhui provinces of China, whose kernel, leaves, and green husks have been widely investigated for nutraceutical components in recent decades (Yang et al. 2015, Jin and Ding 2017). The hickory husks are a byproduct, usually obtained after a cracking and shelling process. The husks are produced in large quantity; they are considered as waste and could create environmental problems. Efforts have been made to extract valuable nutraceutical components, especially from food waste, in recent years (Sha et al. 2016, Belwal et al. 2018). The hickory kernel can be eaten, and shells can be used in preparation of activated carbon; but the husks are not edible so, except for a few of the husks returned to the forest as fertilizer (Zhao et al. 2020, Lu et al. 2022), most of the husks are discarded by farmers at will into rivers, streams, ditches, sewage terminal ponds, and other places, causing serious pollution to the water and soil. Some farmers burn the husks and the husk ash that is produced contains alkali, which pollutes the land to a certain extent. Therefore, it is of great practical significance to study reuse of hickory husks. In this study, optimum technology of forming hickory husks, and its forming mechanism, were analyzed.

Materials and Methods

Experimental instruments and equipment

The test equipment mainly included a hydraulic-plunger forming machine (made by Engineering College of Beijing Forestry University), NI USB-6221 data acquisition card, SC69-02 moisture rapid tester and Leica S8 APO stereo microscope, WV-CP470 digital color CCD camera, vernier caliper, SF-400S electronic scale (0.01 g), portable scale (1 g), sample bag, watering can, Retsch SM 300 pulverizer (Haan, Germany), etc. (Fig. 1). The hydraulic part of the forming machine was a BHD-32 hydraulic system produced by Shandong Hydraulic Machinery Manufacturing Corporation. The system pressure was 25 MPa; the total power was 11 kW; the total flow was 45 L/min. The material compression speed of the piston was 30.6 mm/s; the diameter of the piston was 60 mm; the theoretical maximum thrust was 508.9 kN; the maximum pressure was 180 MPa.

Figure 1.—The structure of a hydraulic briquetting machine with plunger.

A HSLT-PTGV23 pressure transmitter was selected to measure the axial pressure of the material, and the pressure transmitter was directly installed on the hydraulic pipe of the oil inlet channel through a threaded connection. When the hydraulic forming machine was running, the pressure transmitter could measure the pressure of the hydraulic system. A TJH-10 microload sensor was used to measure the radial force on the material at different positions of the forming mould.

Experimental materials

The raw material used was matured, peeled hickory husk collected in autumn 2022 in Lin ’an District, Hangzhou, Zhejiang Province. Hickory husk refers to the green husk on the outer layer of green hickory fruit, not the hard shell on the outside of the nut, which is commonly known as “Guo Pu” in Chinese. After natural drying in the field, the husks were collected and crushed with a Retsch SM 300 pulverizer (Haan, Germany), and sifted to obtain 5-mm sizes. The husks were completely dried with a rapid moisture tester, and total moisture was calculated to be about 6 percent. In addition to the naturally dried husks, it is also necessary to adjust the water content of husks to 10, 14, 18, 22, and 26 percent as test raw materials. Specific treatment process was as follows: (1) adjust the moisture content on the basis of total moisture of natural dried husks, and calculate the amount of water to be added. (2) Put the test raw materials into a plastic bag, spray water with a watering can, stir continuously, seal, and stand at room temperature for 72 hours so that water and materials are fully and evenly mixed. (3) After step 2, measure water content of the husks after standing; and if it did not meet the test requirements, the water content should again be adjusted.

The formula for calculating water content was

$$M = \frac{m - m_0}{m_0} \times 100\% \tag{1}$$

In the formula, M refers to material moisture content, in percent (%); m refers to material weight before drying; m_0 refers to material weight after complete drying.

Experimental method

The test was carried out on a hydraulic-plunger forming machine at an ambient temperature of 18° to 25°C. The plunger was driven by hydraulic cylinder to squeeze the hickory husks intermittently. With the accumulation and compression of the husks in the forming mould, the friction between the material and the inner wall of the mould gradually increased, and the materials were compressed in the mould. The shape and size of the husks were determined without secondary crushing, and it was unnecessary to consider the influence of particle size. Test parameters measured were water content and length–diameter ratio (LDR) of the forming mould. The self-made open forming mould was used in the test. Its inner diameter was 60 mm; lengths were 180, 210, 240, 270, 300, and 330 mm; and the corresponding LDRs were 3, 3.5, 4, 4.5, 5, and 5.5, respectively. Thirty-six constitutive tests were carried out by controlling the water content of the husks and the LDR of the forming mould, respectively.

During the test, after each extrusion of the plunger, materials with roughly the same quality were added to the hopper to ensure that the degree of compressed material was uniform each time; and the pressure change in the oil inlet tube was recorded by the pressure transmitter measurement and data acquisition card, and the pressure exerted by the plunger was converted. The plunger movement stopped at the end of the stroke and was discharged after 10 seconds for the next extrusion.

Data acquisition and processing

In the forming experiment, a TJH-10 miniature load sensor was used to measure the radial force on the material at different positions of the forming mould. The surface of the pressure sensor can sense the change of pressure and convert the pressure value into voltage signal. However, the voltage signal generated in case of small change in the pressure value was weak because of the small force area, so the voltage signal needed to be amplified by the TB3K single-channel transmitter after output. At the same time, the HSLT-PTGV23 pressure transmitter was used to measure the pressure of the hydraulic system in the hydraulic forming machine, and the pressure value was also outputted by voltage signal. The USB-6221 data acquisition card A/D system (American NI Company) was used to collect voltage signal output via two pressure sensors in real time, and the voltage analog signals were controlled, monitored, and saved to the computer via LabVIEW2017 virtual instrument software. In order to reduce external interference, the terminal was configured as grounding reference single-end mode (RSE), and actual sampling rate was 100. The number of samples was 10. According to the corresponding relationship between voltage and pressure, the voltage value can be converted on the computer, and the corresponding pressure value can be obtained. The radial compressive stress of the material at a fixed point of the forming mould was

$$\sigma_F = \frac{F}{\pi r^2} \tag{2}$$

where, σ_F is radial compressive stress on the material (unit: MPa); F is the radial pressure measured by the sensor (unit: N); and r is convex radius (unit: mm).

The pressure value of the hydraulic system of the forming machine was measured by the HSLT-PTGV23 pressure transmitter. It was also necessary to convert the pressure of the hydraulic system into the plunger pressure according to Formula 2, and the converted value was the axial pressure value of the material in the moulding process. The inner diameter of the hydraulic cylinder of the hydraulic forming machine was 180 mm, the front end of the piston rod of the hydraulic cylinder was consolidated with the plunger, and the diameter of the plunger was 60 mm. The pressure exerted by the plunger during the movement can be obtained (i.e., the axial compressive stress of the material).

$$\sigma_P = 9\sigma_S \tag{3}$$

where, σ_P is axial compressive stress of the material (unit: MPa); and σ_S is hydraulic system pressure (unit: MPa).

Table 1.—Factors and levels of test.

	Experimental factors	
Level	Moisture content (%)	Length–diameter ratio
1	6	3
2	10	3.5
3	14	4
4	18	4.5
5	22	5
6	26	5.5

Experimental design

Different raw materials have a variety of structures and component contents, so biomass briquette fuel requires the use of different pressures and temperatures during the compression moulding process (Carone et al. 2011). In addition, the physical properties of raw materials and the LDR of forming mould will affect the moulding process. In order to verify the moulding mechanical laws of hickory husks under different moulding conditions, the moisture content of the raw materials and LDR of forming mould were used as experimental factors, and the levels of experimental factors are shown in Table 1.

Thirty six groups of single-factor experiments were carried out on hickory husks to measure the axial and radial pressures of raw materials forming under different experimental factors and levels, and the values were measured multiple times under the same factor level (Table 1). Three groups of pressure values under stable forming conditions were taken and their average values were calculated, which was used as the actual measured pressure.

Relaxation density of biomass moulding fuel

Relaxation density and surface quality of the biomass moulding fuel were used as indices to evaluate the influence of different forming conditions on the forming quality, which was used as the basis for determining forming

Figure 2.—Four different surface-quality categories of biomass moulding fuel (BMF) briquettes.

conditions of biomass raw materials. After moulding, the compression density of the BMF briquette gradually decreases as a result of elastic deformation and stress relaxation, and the density of the briquette became stable after a certain period of time. At this time, density of the BMF briquette was called relaxation density. After each group of tests, three rod-shaped moulding blocks were randomly selected and their mass, length, and diameter were measured after being placed in the natural state for 72 hours, and the average relaxation density was calculated to evaluate the moulding quality.

In order to study the influence of each experimental variable on the moulding quality of the BMF, the density and diameter of the BMF were used. Pressure resistance and surface quality of the BMF were used as an evaluation index of the forming quality. To obtain the density of the BMF, quality of the BMF was measured using an electronic scale (model: SF-400) and the vernier caliper was used to measure diameter and length of the BMF. To ensure the reliability of results, multiple locations were selected during measurement and the measurement junctions were combined.

Take the mean value and calculate the fuel density according to the formula

$$\rho = 4m/\pi D^2 L \tag{4}$$

where ρ is the density of moulded fuel (unit: g/cm^3); m is the weight of the sample of moulded fuel taken (unit: g); D is the diameter of the moulded fuel sample (unit: cm); and L is the length of the moulded fuel sample taken (unit: cm).

Observation of microstructure of BMF

Evaluation of the surface quality of moulded fuel was mainly based on the number of surface cracks. For convenience, to check processing and analysis of the data, surface quality of the BMF briquette was quantified and the processing method was as follows: (A) poor moulding quality; (B) more cracks on the surface of BMF briquette; (C) fewer cracks on the surface of the BMF briquette; and (D) smooth surface of the BMF briquette.

The test samples were placed on the stereo microscope stage, and the microscopic structure of the end face of the shaped block was photographed by the camera. The bonding state at the boundary of the husks was observed, and the forming state of the husks under different conditions was analyzed from the microscopic point of view.

Results and Discussion

Different quality of the BMF briquettes

The BMF briquette obtained by hydraulic-plunger moulding was formed by a number of forming pieces combined with each other, and each movement cycle of the plunger compressed the materials into a small forming block. The plunger produced intermittent compression of

Table 2.—Experimental results of compression briquetting. BMF is biomass moulding fuel.

Moisture content (%)	Length–diameter ratio	Extrusion or not	Quality of the BMF	Relaxation density ($g \cdot cm^{-3}$)
6 (natural drying)	3	Y	N/A	—
	3.5	Y	A	0.40
	4	Y	A	0.42
	4.5	Y	B	0.52
	5	N	—	—
	5.5	N	—	—
10	3	Y	N/A	—
	3.5	Y	B	0.52
	4	Y	B	0.54
	4.5	Y	C	0.69
	5	Y	C	0.56
	5.5	N	—	—
14	3	Y	N/A	—
	3.5	Y	C	0.76
	4	Y	C	0.78
	4.5	Y	D	0.98
	5	Y	C	0.82
	5.5	Y	N/A	—
18	3	Y	N/A	—
	3.5	Y	N/A	—
	4	Y	B	0.56
	4.5	Y	B	0.56
	5	Y	C	0.67
	5.5	Y	N/A	—
22	3	Y	N/A	—
	3.5	Y	N/A	—
	4	Y	N/A	—
	4.5	Y	B	0.55
	5	Y	B	0.56
	5.5	Y	C	0.68
26	3	Y	N/A	—
	3.5	Y	N/A	—
	4	Y	N/A	—
	4.5	Y	N/A	—
	5	Y	B	0.54
	5.5	Y	B	0.59

the material, and the hydraulic system efficiency was low; therefore, compression interval time was long and a border crack appeared between the adjacent forming blocks. However, better forming conditions improved the state of the crack. When the moisture content was constant, the forming quality of the material could be improved by appropriately increasing the length–diameter ratio (LDR). Figure 2 shows the forming quality of hickory husks under different forming conditions. The surface quality of the BMF was divided into Figure 2(a), (b), (c), and (d), respectively. Figure 2(a) illustrates that the forming quality of the BMF was worse with more cracks on the surface of the BMF. Figure 2(b) illustrates that the forming effect of the BMF was better with fewer cracks on the surface of the BMF. Figure 2(c) illustrates that the fuel surface was smooth with fewer cracks. Figure 2(d) illustrates that the fuel surface was smooth without cracks. These were converted to A, B, C, and D, respectively, in alphabetical order, and the experimental results are presented in Table 2.

Test forming result

The data associated with different experimental conditions of compression briquetting were differentiated, and the water content, LDR of the forming mould, and relaxation density of BMF were obtained. The experimental data of degree and surface quality of BMF are shown in Table 2.

The influence of experimental parameters on moulding pressure

In the moulding process, the raw materials go through the compression stage, compaction stage, and moulding stage, and the pressure of the materials at each stage is different. Through the measurement of axial pressure by the pressure transmitter, the pressure change curve of the plunger compression materials could be obtained. When the plunger moved to the end of the stroke, the thrust was greater than the maximum pressure of the hydraulic system, resulting in overflow of the relief valve and maximum pressure value of the curve tending to be 160 MPa. Figure 3(a) shows the influence of LDR of forming mould on moulding pressure under different moisture contents. With the increase of LDR, the pressure required for moulding raw materials showed an upward trend. As the LDR of the forming mould increased, the materials in the mould increased, friction between the materials and the inner wall of the mould increased, and the moulding pressure required to overcome the friction increased accordingly. The moulding pressure was too large and the moulding energy consumption increased correspondingly, which was not conducive to actual production. Figure 3(b) shows the influence of water content on moulding pressure under different LDR of the forming mould. With the increase of water content, moulding pressure presented a trend of first increasing and then decreasing. When the moisture content was low (<14%), moulding pressure increased with the increase of the moisture content. At this time, the moisture acted as an essential free radical in the BMF, which played a bonding role; and the combination between the particles of raw materials became closer, which increased friction between the materials and increased the peak pressure. When the moisture content was high (>14%), moulding pressure decreased with increase of the moisture content. At this time, too much water formed a water film around the raw materials, preventing proximity of the raw material particles and combination of particles. At the same time, friction between the material and the inner wall of the forming mould would be reduced, making the moulding pressure smaller.

Effect of test parameters on relaxation density

Relaxation density was an important index to measure physical quality characteristics of BMF. Figure 4(a) shows the influence rule of LDR on relaxation density under different water content. With the increase of LDR of the forming mould, relaxation density showed a trend of first increasing and then decreasing. When the length diameter of the forming mould was small, friction

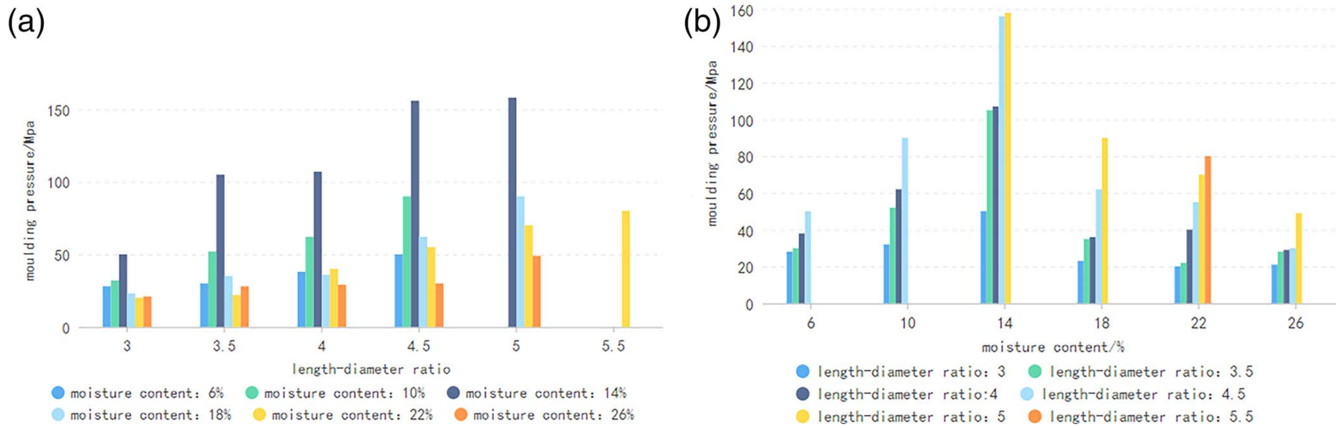

Figure 3.—Effects of test parameters on moulding pressure.

between the material and the inner wall of the mould and the materials was small, binding ability of adjacent materials was small, and the BMF briquette was loose. When the LDR of the forming mould increased, forming pressure increased, bonding ability between materials increased, and density increased. However, when the LDR of the forming mould was too large, a large amount of air was added to the materials in the extrusion process, and the water between the materials was vaporized into water vapor as a result of friction heat. During compression, air and water vapor accumulated in the forming mould and could not be discharged in time, and they formed air pockets in the moulding fuel. At the moment when the BMF briquette was out of the mould, high pressure gas was suddenly released, resulting in the cracking of the BMF briquette and even the phenomenon of "shooting (Excessive steam pressure caused the biomass briquette to explode into several sections.)," which greatly reduces the density of the BMF briquette. Figure 4(b) shows the influence of water content on relaxation density under different LDRs of forming mould. Under the same LDR, the relaxation density presented a trend of first increasing and then decreasing with the increase of water content. If the moisture content was too low, the small amount of water in the material was not enough to play the role of binder, contact area between the particles was small, and the material was not easy to combine. If the moisture content was too high, the moisture content of the corresponding BMF briquette was high, it was easy to expand after the mould, and would become loose as a result of water evaporation in the natural environment.

Microstructure of BMF briquette under different LDRs

Figure 5 shows the microstructure of the end face of the BMF briquette under different LDR of the forming mould when the water content was 14 percent. The hickory husks still maintained their original flake shape under the stereomicroscope because they had not been crushed twice. When the LDR of the forming mould was 4, the boundary of the material was relatively clear, the flakes were stacked with each other, the bonding phenomenon was not obvious, and there were small gaps indicating that the moulding pressure was small. In that case, the extrusion effect was not enough and the material was not completely compacted. When the LDR of the forming mould was 4.5, the materials were compacted and chimeric, the combination was the closest, and the stacking phenomenon was not obvious. When the LDR of the moulding sleeve was 5, the boundary between the materials was clear and the gap was large because the moulding pressure was too large, the BMF briquette expanded after moulding, and the residual internal stress caused the material to rebound. It can be concluded that the LDR of forming mould deeply affected the bonding

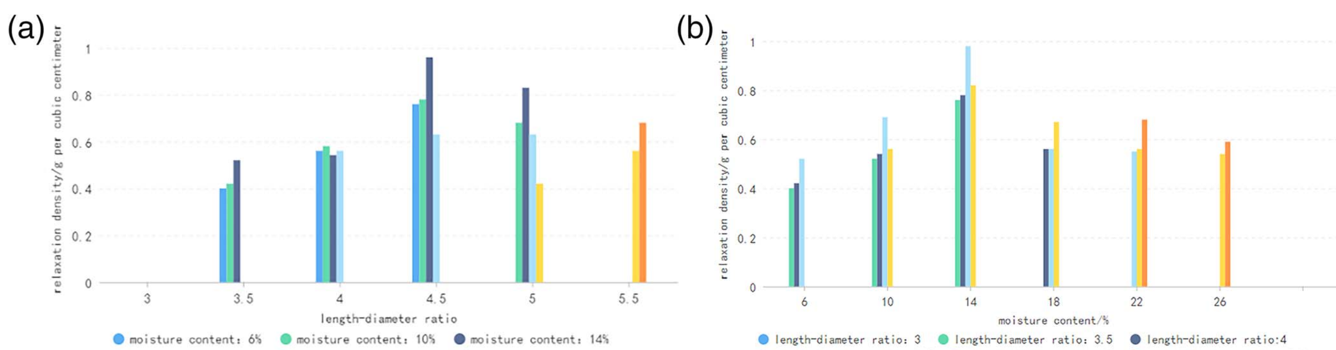

Figure 4.—Effects of test parameters on relaxation density.

Figure 5.—Microstructure of briquettes under different length–diameter ratios.

strength between materials on the micro level, thus affecting the macro forming effect.

Microstructure of BMF briquette under different moisture content

Figure 6 shows the microstructure of the end face of the BMF briquette under different water content when the LDR of the forming mould was 4.5. When the moisture content of hickory husks was 6 percent, there was warping phenomenon, there was a large gap between the materials, and the stacking phenomenon was obvious. A small amount of water vapor in the raw materials was not conducive to the rapid conduction and uniform distribution of heat; therefore, it was not easy to reduce the softening point of lignin, the bonding effect was not large at lower temperatures, and the particles were not sufficiently extended and could not be closely combined. When the moisture content was 14 percent, the materials were extruded from each other, the lamination phenomenon was not obvious, and the bonding effect was the best. The increase of water caused the materials to became softer, flatter, and more ductile; and a small amount of water adsorbed on the surface could reduce the roughness, increase the contact area between the materials or reduce the spacing, and thereby increase the weight of the BMF briquette. When the moisture content was 22 percent, the distance between the materials was larger, and the combination did not occur. Too much water increased the distance between the material particles, reduced the attraction between particles, and led to an increase in water vapor, occupying more space.

Conclusions

In this study, the compression densification moulding process of hickory husks was carried out to investigate moulding pressure, relaxation density, and surface quality using a hydraulic-plunger forming machine with different moisture contents of hickory husks and different length–diameter ratios (LDR) of the forming mould. Based on the experimental results, it can be concluded that the moisture content of the raw materials and LDR of the forming mould significantly influenced moulding pressure, relaxation density, and surface quality of the BMF. With an increase of the LDR of the forming mould, the average moulding pressure and relaxation density will increase with the same moisture content. In addition, with an increase of the moisture content of the raw materials, the average moulding pressure and relaxation density will increase first and then decrease with the same LDR of the forming mould. BMF compressed with a relatively high and low moisture content of the raw materials will have many cracks on the surface, and may even break into small pieces during production. As a result, a relatively high or low moisture content is not favorable for densification moulding. When the water content of raw materials is 14 percent and the LDR of the forming mould is 4.5, the moulding quality is the best and the relaxation density of the BMF is 0.98 g/cm^3. By observing the microscopic structure of the end face of the BMF with a stereomicroscope, it is found that when the moisture content is 14 percent and the LDR of the forming mould is 4.5, the bonding state between the materials is the best, which is consistent with the macroscopic forming effect. Experiments show that a hydraulic-piston forming machine can utilize hickory husks to produce biomass moulding fuel at room temperature, which not only expands the application range of hickory husks, increases farmers' income, and reduces environmental pollution, but also helps to achieve carbon neutrality and carbon peak.

Figure 6.—Microstructure of briquettes under different moisture content.

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