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Research article

Parametric studies on energy utilization of the Chinese medicine residues: Preparation and properties of densified pellet

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ABSTRACT

The depletion of fossil fuels has fueled an increased interest in biomass resources usage for heat and electricity generation. As an important biomass resource, Chinese medicine residues have great potential in substituting fossil fuels. However, that is basically limited by its poor properties, including low bulk density, high moisture content, and inhomogeneous structure. Herein, a safe and sustainable strategy was reported to prepare a high-quality densified pellet derived from Chinese medicine residues to address these worries. In this process, mixed and simple size materials were densified under various moisture content and pressure using a laboratory electronic tablet press machine equipped a single pellet mold. Results showed that higher pressure, ideal moisture content (~6.5 %), and mixed particle size could densify better quality pellets. These findings pave the way for the safely and efficient resource utilization of Chinese medicine residues, as well as providing theoretical guidance and technical support for the household heating.

1. Introduction

Over the last few decades, fossil fuel consumption has risen with the rapid growth of the world population, resulting in negatively environmental impact and climate change [1]. For the past period of time, considerable efforts have been made to develop environmentally friendly alternatives to limit the pace of climate change and avert an energy catastrophe. Considering such solutions, biomass is the most promising energy source for attaining carbon peaking and carbon neutrality goals. Extensive research has showed its capacity to reduce greenhouse gas emissions and saving non-renewable resources [2].

Chinese medicine residues (CMRs) refers to the solid waste produced in the processing, e.g. steaming or chemical extraction, of Chinese medicine materials. Up to now, its production has reached 60–70 million tons every year [3]. Typically, traditional disposal methods for the CMRs including incineration, landfill, burial, and so on [4,5]. It not only seriously pollutes the environment and causes a huge resource wastage, but also seriously endangers human health [6]. Hence, how to effective use CMRs and develop a sustainable, economical, environmentally friendly method to reduce their environmental hazards and turn waste into treasures has become a very urgent and significant work.

Nevertheless, the direct utilization of CMR is constrained by its inferior physicochemical properties. Furthermore, poor bulk density can also cause irregular temperature distribution, resulting in low combustion efficiency [7]. To address the above crisis, densification, as an environment friendly and higher processing capacity attractive method to minimize these worries, has gained worldwide

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attention [8]. Densification refers to densify biomass to high-density energy carriers with regular shape and size [9]. Moreover, it minimizes costs per unit raw materials transported and storage, and improves its combustion efficiency in heating or energy generation [10,11].

The primary determinants of the densified pellets quality for further handle and storage are their density and durability. Till date, relevant studies mainly focused on how the pellet quality properties are affected by processing factors. Moisture was the primary determinant for density and mechanical strength, as well as influence net calorific value and combustion efficiency of made pellets [12]. In addition, results obtained by Navalta et al. (2020) indicated that pressure has an obvious influence on the briquette density, and the density and pressure was directly proportional [13]. It would be interesting to discuss the effect of particle size on pellet quality. As study presented by Pradhan et al. (2021) fine feedstock lowered production costs by 23 % compared to that of coarse [14]. To conclusion, these researches agree that the ideal parameters are mostly determined by the sort of raw materials used. However, no studies were ever conducted on CMRs to determine the association between pelletization parameters and pellet quality.

Hence, with the current study, CMRs were employed as a raw material to prepare densified pellets. Quality characteristics that include density, durability (Meyer hardness), and combustion performance were evaluated. Our objective is to develop the usefulness of CMRs for processing into fuel pellet and provide an insight into bonding mechanism. The concurrent purpose is to provide theoretical suggestions for domestic heating, as well as an effectively resource utilization of CMRs.

2. Materials and methods

2.1. Materials

The used CMRs were supplied by Rongchang Pharmaceutical, Ltd. in Zibo city, Shandong province, China. The collected materials were firstly air dried for a couple of days under sunlight conditions. After that, they were ground to a particle of less than 0.4 mm and then sieved using vibrating screening machine with various mesh size screens (e.g., 40, 60, 80, 100, 120, and 140 mesh). The as received materials were further dried, cooled, and then kept in a sealed plastic container.

Proximate analysis was measured with an industrial analyzer (SDTGA-8000, Sundy, China). The elemental contents were determined using an elemental analyzer (Vario EL Cube, Elementar, Germany), which the oxygen (O) contents were calculated by difference [15]. The higher heating values (HHV) were calculated using the following equation [16]. All of the results are shown in Table 1.

$$\mathrm{HHV} = -4.914 + 0.4114\mathrm{C} + 0.6114\mathrm{H} + 0.2611\mathrm{N} + 0.3888\mathrm{S} + 0.02097\mathrm{O}\ (\mathrm{MJ/kg})$$

2.2. Adjustment of moisture content

According to the experimental design, the dried CMR was humidified to ~6.5 %, ~11.5 %, ~16.5 %, and ~21.5 % using a sprayer. For the experiment, raw materials were placed evenly in a tray. After that, the corresponding mass of deionized water was calculated by the following equation (1), and then sprayed on the raw materials for homogeneous mixing. Subsequently, they were filled in a sealable plastic bag and stored before further analysis.

$$m_{add} = m_{ini}\frac{W_2 - W_1}{1 - W_2} \tag{1}$$

where, m_{add} is the added deionized water mass (g), m_{ini} is the initial mass before adjustment (g), W_1 is the initial moisture content (%), and W_2 is the target moisture content (%).

2.3. Densification process

Pellets were prepared using a laboratory electronic tablet press machine (JDP-20S, Jingsheng Scientific Instrument Co. Ltd., Shanghai, China) with a single pellet mold. The mold is consisting of a plunger with 19.8 mm in diameter and 40 mm in height and a cylinder die with a diameter of 20 mm and a length of 30 mm. Detailed densification process is illustrated in Fig. 1. Briefly, ~0.5g of material was weighted and placed in the mold. After that, the pelletization was conducted at a pre-determined pressure (i.e., 120 MPa, 180 MPa, 240 MPa, 300 MPa, and 360 MPa). The compaction was carried out without heating and any binder. After the set compression pressure was reached and held for 30 s. Then the pellet was taken out and stored.

Table 1
Properties of Chinese medicine residue sample.

Proximate analysis (%)				Ultimate analysis (%)					Heating value (MJ/kg)
Moisture content	Volatile atter	Fixed carbon	Ash	C	H	O[a]	N	S	
1.25	67.69	15.75	16.56	50.27	6.84	40.91	1.72	0.26	21.36

Note.
[a] Calculated by difference.

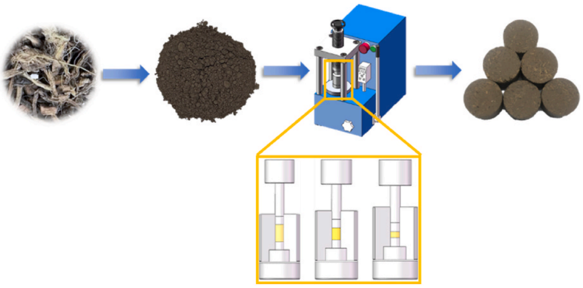

Fig. 1. Schematic of densification process.

2.4. *Characterization of pellet*

2.4.1. *Density*

After 48 h, the mass, length and diameter were recorded to calculate density by using the following equation (2) [17]. The detailed mass, length, and diameter of the pellets are presented in supplementary material. Each group was tested at least 3 times, and the test results were averaged.

$$D_e = \frac{4m}{\pi d^2 l} \tag{2}$$

where, D_e is the pellet density (g/cm^3), m is the pellet mass (g), d is the diameter (cm), and l is the length (cm) of pellet, respectively.

2.4.2. *Meyer hardness*

Hardness is one of the important properties of pellets, which could reflect the possibility of breakage during handling, transportation, and storage. For the present study, pellet was horizontally placed on the bottom fixed plate. After that, the upper moved plate was run at a speed of 5 mm/min until the pellet was broken, and then the maximum load was recorded [18]. The Meyer hardness (H_m) is defined as the load divided by the projected indentation area and calculated using the following formula (3) [19].

$$H_m = \frac{F}{\pi\left(Dh - h^2\right)} \tag{3}$$

where, H_m is the Meyer hardness (N/mm^2), F is the maximum load (N), D is the pellet diameter (mm), and h is the indentation depth (mm).

2.4.3. *Combustion characterization*

Combustion characterization of the pellets was evaluated by the thermogravimetry analysis (TGA). TGA is one of the most common techniques and has attracted great attention in understanding combustion characteristics of fuel pellet [20–24]. The combustion experiment was conducted using thermogravimetric analyzer (STA 449 F5, Netzsch, Germany). Approximately 10 mg sample was used and heated from 30 to 1000 °C with a heating rate of 10 °C/min with airflow rate of 50 mL/min.

3. Results and discussion

3.1. *Physicochemical properties of the Chinese medicine residue*

Table 1 shows the findings of proximal and ultimate analysis. It is worth noting that moisture content adversely affects the heating value, for part of the energy produced is needed for the evaporation of water during combustion [25]. However, it is not the case that the lower moisture content is better for biomass pelletization. Lei et al. (2019) pointed out that water can play an important role in transferring pressure, reducing friction between particles, and homogenizing temperature field in the formation of pellets [26]. Proximate analysis can help to assess the combustion characteristics of biomass. The proportions of volatile matter and fixed carbon are critical for the heating value, as well as the thermal utilization. Based on the results, 67.69 % of volatile matter and 15.75 % of fixed carbon, it can be considered as fuel for generating heat and power [27]. Moreover, a higher ash content in biomass raw material may cause ignition and combustion problems, along with environmental pollution issues. The ash content obtained was 16.56 %, which was much higher than other biomass that was found in relevant literature [27–29]. This may cause by raw material collection process. After steaming, the CMR was transported to the open area for the sun-dried, leading to the addition of soil and sand, hence result in an increase.

The ultimate analysis is a chemical analytical methodology for investigating the various chemical ingredients. The result showed that the CMR biomass has a higher carbon (50.27 %), hydrogen (6.84 %), and oxygen (40.91 %), while a lower nitrogen (1.72 %) and sulfur (0.26 %). The amount of carbon and hydrogen is useful in contributing the quality of the combustibility, for the hydrogen to the carbon ratio [30]. The oxygen content is higher than that of coal, enabling combustion with less air demand than with coal. The extremely lower sulfur content is part of the advantages of fuel purpose, which will significantly reduce SO_2 emissions and consequently reduce environmental problems such as acid rain.

3.2. Impact of moisture content on pellet density and Meyer hardness

Moisture content is a vital parameter in the production of densified pellet. Table 2 display the calculated pellet density and Meyer hardness of CMR pellet prepared with different amount of moisture. Density initially increased to the maximum (1382.45 kg/m^3) with increasing moisture content from ~1.25 % to ~6.5 %, and then decreased. Nevertheless, it is still higher than the grade in the standardized quality classification (>1000 kg/m^3). As evident from Table 2, the Meyer hardness showed a similar trend, also peaking at a moisture content of ~6.5 %. Hence, an optimal moisture content of ~6.5 % was suggested for pelletizaiton of Chinese medicine residues. This conclusion was consistent with prior research that showed that pellet quality (e.g., density, durability) had a peak value at the optimal moisture content [31]. Higher or lower moisture content will be detrimental to the pelletization, resulting in poor quality pellets. This had also been verified in the present study, as shown in Fig. 2, which depicted the cracks under higher moisture content.

In view of report of Rumpf, the binding mechanisms were classified to five categories, namely solid bridge, adhesive and cohesive forces in non-freely-movable binders, interfacial forces and capillary pressure at freely-movable liquid surfaces, attractive forces between particles (van der Waals's or electrostatic forces), and interlocking bonds [32]. Generally, they are caused by water to a greater or lesser extent. Water generates a series of chemical changes with heat, such as reducing the glass transition temperature of lignin, denaturing proteins, and gelatinizing starch, which improves the solid bridge as well as adhesive and cohesive forces [33]. Water functions as a film binder, strengthening and enhancing bonding through van der Waals forces by increasing particle contact area. A thin layer of water around the particles would form bonds by capillary sorption [33]. Furthermore, water can minimize particle friction, allowing particles to flow more freely and increasing the mechanical interlocking effect [34].

In this study, the diffusion ability of water is weakened when the moisture content is lower than ~6.5 %, leading to the poor fluidity of material particle, as well as the particles are unable to extend smoothly. An appropriate moisture content is also necessary to reduce the friction of particle-particle. Under the moisture content of ~6.5 %, the existence of water promotes the mobility of small particle size to eliminate the pore space, thus increasing the density and Meyer hardness. Not only that, the presence of water could also contribute to decrease the glass transition temperature of lignin [30]. Upon this optimal moisture content, the solid bridge between particles were improved by the softened lignin, resulting well bonding. While in the higher moisture content (beyond ~6.5 %), the water tended to form a barrier layer around material particle, which hindered the combination or particles, also limit the release of natural binders from the materials [35]. This is the evidence for the decrease of pellet quality, as well as the reason for pellet fracturing.

3.3. Impact of pressure on pellet density and Meyer hardness

The effect of pressure on density and Meyer hardness was investigated by applying various pressure (i.e., 120 MPa, 180 MPa, 240 MPa, 300 MPa, and 360 MPa). As shown in Fig. 3, the density increased continuously as the pressure went up. It was improved from 1300.22 to 1359.77 kg/m^3 when pressure was increased to 240 MPa, contributing to a 4.5 % increment. That was much higher compared to the 1.5 % increase while applied pressure further raised to 360 MPa. In the report of Lei et al. (2019), they pointed out that there will be three stages during compression. The physicochemical characteristics (e.g., density) increase rapidly as the pressure rises in the previous two stages, while the quality index keeps a lower growth under the third stage [26]. Akin to how pressure affects pellet density, the Meyer hardness further confirmed that pressure is limited to pellet quality improvement (see Fig. 3).

The natural binding components in CMR material are squeezed out under pressure, which contributes to making solid bridges [36]. As an example, the macromolecules of lignin are susceptible to fragmentation, then condensed and degraded, resulting in soluble lignin and insoluble lignin. Furthermore, the existence of phenolic hydroxyl and alcoholic hydroxyl promotes the dissolution of alkaline lignin. Lignosulfonate dissolves with water to form a colloidal solution that works as a binder and improves the binding strength by adhering to and polymerizing particles. The concrete manifestation is the increase of density and Meyer hardness. In theory, the higher the pressure applied, the better quality can be obtained. However, it is actually not the case due to the limitation of cost and other issues. Therefore, the appropriate pelletization pressure for Chinese medicine residues is 240 MPa.

From a different perspective, particles are in motion slowly under lower pressure in the initial stage of pelletization, and may not be able to overcome friction between wall and particles, exhibiting relatively lower density and Meyer hardness. Nevertheless, some of the interparticle air and water were still expelled. Simultaneously, some particles occupy the pore space under the promotion of pressure. With the continuous action of pressure, particles undergo a phenomenon of position dislocation, or, in other words, the

Table 2
Pellet density and Meyer hardness at various moisture content.

Moisture content (%)	Density (kg/m^3)	Meyer hardness (N/mm^2)
1.25	1333.42 ± 7.79	3.87 ± 0.42
6.5	1382.45 ± 2.10	8.59 ± 0.07
11.5	1362.41 ± 0.92	7.82 ± 1.00
16.5	1318.57 ± 5.98	4.09 ± 0.39
21.5	1285.97 ± 4.03	1.93 ± 0.27

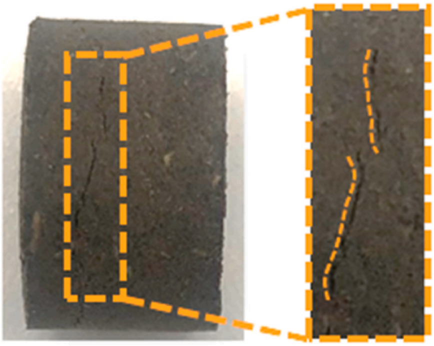

Fig. 2. Cracks under higher moisture content pelletization.

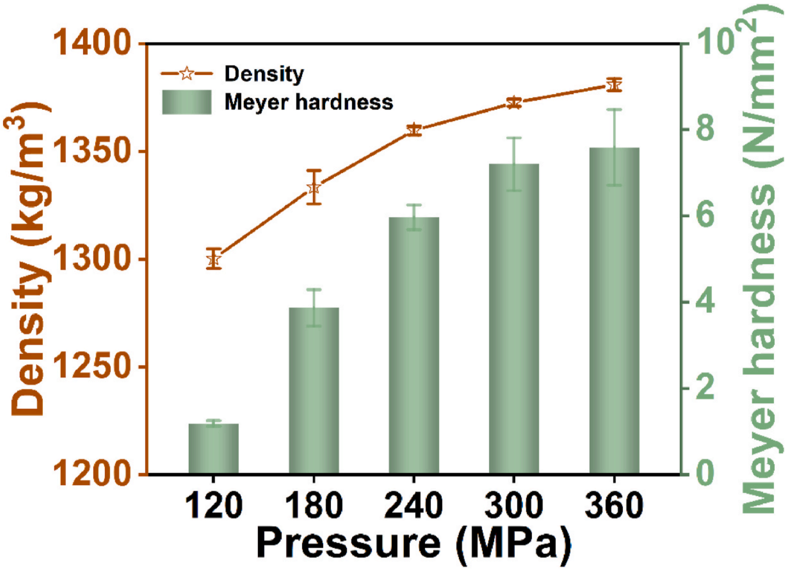

Fig. 3. Effect of pressure on pellets density and Meyer hardness.

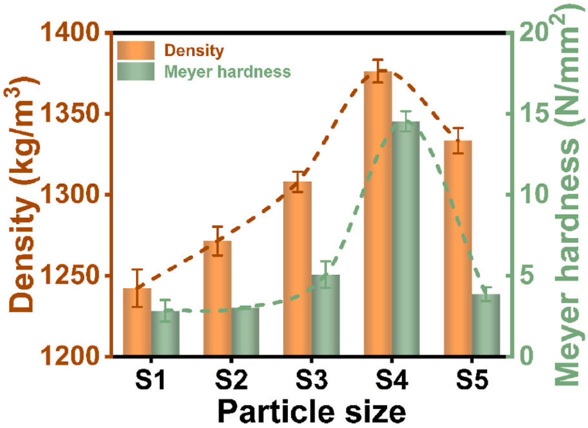

Fig. 4. Effect of particle size on the density and Meyer hardness (S1: 60–80 mesh, S2: 80–100 mesh, S3: 100–120 mesh, S4: combination of 40–60 mesh and beyond 40 mesh, and S5: full-size).

original disorderly arrangement gradually turns to orderly. Along with the compression proceeding, the inter-particle gap gets smaller, at which time the large sized particles are broken into fine particles and deformed to fill the narrower gaps between the particles. When the pressure is further increased, the particles are plastically deformed, as well as extended in the direction perpendicular to the principal stress, resulting in the neighboring particles being in close contact with one another through mechanical interlock [26]. Particles become thinner and adjacent particles are in close contact by bonding parallel to the principal stress direction. As the biomass is an elastic-plastic body, it will not recover to its original structure and morphology after plastic deformation, and some residual stress will be stored among the particles, which will make the particle combination stronger. This may be an important evidence for the well compactness of pellet.

3.4. *Impact of particle size on pellet density and Meyer hardness*

In the present study, two mixed materials (The first is a combination of 40–60 mesh and beyond 140 mesh. The second is the crushed and unscreened raw materials, which marked full-size materials) and three simple size materials (60–80 mesh, 80–100 mesh, and 100–120 mesh) were chosen to investigate the impact on pellet density and Meyer hardness. For convenience, 60–80 mesh, 80–100 mesh, 100–120 mesh, the combination of 40–60 mesh and beyond 40 mesh, and full-size materials were replaced by S1, S2, S3, S4, and S5, respectively.

As shown Fig. 4, both the quality parameters increased with the decreasing particle size. There are two main hypotheses for the influence of particle size on pellet quality. First, higher particle sizes provide more gaps and weak van der Waals forces, limiting particle interlinking and resulting in lower-quality pellets. Second, small particle size results in a large surface area of contact, which facilitates the formation of strong interparticle bonds, leading to a densely packed structure [11]. However, although milling the feedstock into finer particles favors the production of high-quality pellets, it also increases the cost of grinding [37]. Hence, the particle size becomes a decisive factor in the tradeoff between grinding, pelleting and cost.

It should be noted that the combination of 40–60 mesh and beyond 140 mesh raw material resulted in the highest density and Meyer hardness. It is generally accepted that the gap between larger particle sizes is completely taken by smaller particles in discontinuous particle size systems. Jiang et al. (2014) proposed that, finely powdered materials generate pellets with great density and durability [36]. In this paper, the small particles (less than 0.1 mm) filled the pore space during the densification process. Hence, the volume remains unchanged and the mass continues to rise, leading to an improvement in density and Meyer hardness.

What is interesting in this figure is the decreased density and Meyer hardness obtained in the full-size materials. Normally, full size materials, referred to continuous particle size system, is composed of particles with uniform particle size distribution. During the pelletization process, the gaps between the largest particle sizes are filled by the second-level particle sizes, and then the secondary interparticle gaps are occupied by the smaller particle size, and so on. Hence, theoretically, the maximum density can be achieved under this system. In the present study, the experimental result showed a difference from expectations. It was, on the one hand, perhaps related to the use of small-scale equipment for laboratory use. In addition, it may be caused by the pressure acting on the materials for a short time, leading to the particles cannot able to move, extension, and fill better. Nonetheless, in practice, there is a continued need for full-size materials (i.e., continuous particle size systems) for the preparation of Chinese medicine residue pellets.

3.5. *Combustion characteristics*

For a more intuitive evaluation of the combustion characteristics of CMR pellets, two typical biomass pellets, corn straw (CS) and yak manure (YM) were used to conduct comparative experiments. The detailed information of CS and YM, as well as their pelletization process can be found in previous literature [38,39]. The TG and DTG curves in Figs. 5 and 6 provided a comprehensive comparison of combustion behavior between three pellets. Similarly, all three pellets mainly had three stages, namely dehydration, releasing and combustion of volatile matter, combustion of fixed carbon and burnout. In the first water evaporation stage, it was mainly the loss of

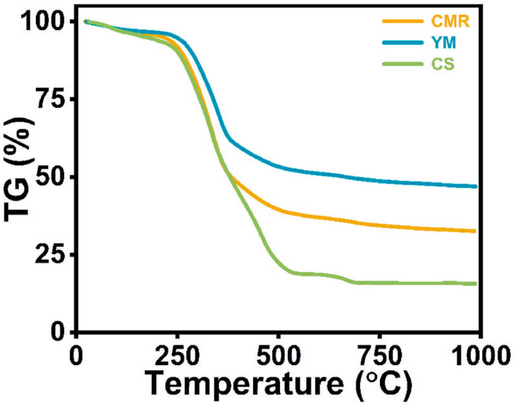

Fig. 5. TG curves of pellets with different kind of biomass (CMR: Chinese medicine residues, YM: Yak manure, CS: Corn straw).

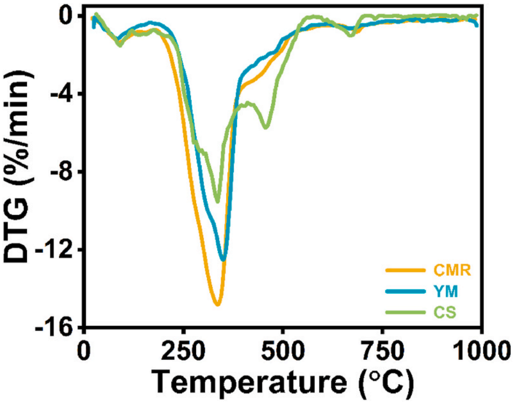

Fig. 6. DTG curves of pellets with different kind of biomass (CMR: Chinese medicine residues, YM: Yak manure, CS: Corn straw).

free and bound water, and a small number of volatiles. The second stage (150–550 °C), attributing to the thermal decomposition of hemicellulose and cellulose. During this stage, large amount of volatile matters decomposed to simple structure and small molecular weight substances. The last stage was referred to combustion of char. From Fig. 5, it was clearly seen that the CMR pellet gave a higher percentage of remaining residues after combustion. This may be caused by the other non-combustible impurities as discussed in **Section 3.1**. The DTG profiles reflect the thermal stability and composition change of pellets. As illustrated in Fig. 6, it has been observed that the temperature of DTG_{max} of CMR pellets shifted to left, representing a lower temperature. In the previous work of Liu et al. (2014), they reported that the value was inversely proportional to reactivity [40]. Thus, the Chinese medicine residue pellets had higher reactivities compared to the other pellets.

The obtained combustion characteristic parameters are summarized in Table 3. Where, the ignition temperature (T_i), burnout temperature (T_f), as well as the combustion characteristic index (S_N) were calculated as mentioned in our previous study [38]. As shown in Tables 3 and it can be found that CMR pellet had lower T_i and DTG_{max}, indicating that it combusted in a more temperate manner. In addition, lower temperature is associated with high pollutant emissions. Therefore, with the appropriate temperature range of CMR pellet, it would be achieved to reduce pollutant emissions by the use of CMR pellet. The combustion characteristic index was used to assess the combustion property, and a larger value indicated better combustibility. In comparison of YM and CS pellets, the combustion characteristics index of CMR pellet increased to 4.87, indicating better combustion performance. Thus, the CMR pellet can be regarded as potential fuel for future household heating.

3.6. Limitation

As evidenced by the present study, Chinese medicine residues could be prepared to densified pellet safely and efficient. However, there are some questions to be further considered. Firstly, the present study was conducted to prepare densified pellet at much higher pressures. However, pellets prepared at higher pressures may affect combustion. In subsequent studies, an appropriate reduction in pressure should be considered while maintaining quality characteristics. In addition, combustion experiments should not be limited to laboratory conditions. For example, a combustion reactor suitable for densified pellet should be developed to monitor the relevant combustion performance online in real time.

4. Conclusions

Results showed that densification is an innovative, effective, and sustainable method for converting Chinese medicine residues into potential fuel. An appropriate moisture content is necessary to reduce the friction and improve solid bridge of particles. The results of the density and Meyer hardness show that excellent pellets can be prepared at moisture content of ~6.5 % moisture. Pressure can promote the position dislocation of particles to fill particle gaps and facilitate mechanical self-locking. Increases in pressure could enhance pellet quality, while the further increase was limited after the pressure reached 240 MPa. The particle size has effective influence on physical properties, especially mixed particle sizes. The reason for this is that the gaps created by large particle sizes can be well filled by small particle sizes, resulting in a relatively dense structure. Combustion characteristics further showed that Chinese medicine residue pellet can be regarded as potential fuel compared to other two residues (yak manure and corn straw).

Data availability statement

Data will be made available on request.

Table 3
The combustion characteristic parameters for pellets.

Pellet	T_i (°C)	T_f(°C)	DTG_{max}(°C/min)	S_N (10^{-8})	Residual mass (%)
CMR	254	409	14.79	4.87	32.56
YM	273	412	12.51	2.92	46.92
CS	255	445	9.54	2.85	15.69

CRediT authorship contribution statement

Jianbiao Liu: Writing – original draft, Methodology, Investigation, Data curation, Conceptualization. **Tianhao Li:** Writing – review & editing, Methodology, Data curation. **Tingting Liu:** Writing – review & editing, Data curation. **Hongzhen Cai:** Writing – review & editing, Funding acquisition, Conceptualization.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.heliyon.2024.e36947.

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