



# Pelletization Characteristics of the Hydrothermal Pretreated Rice Straw with Added Binders

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## Abstract

Pelletization of the loose rice straw is an attractive option to produce renewable fuels. In this paper, we focus on the problem of how to improve this pelletization process, especially reduce energy consumption and improve product quality. In detail, we pretreat rice straw and investigate the densification characteristics of the pretreated materials. Pretreatment methods of the materials include hydrothermal treatment and adding a certain proportion of economic additives, such as rapeseed meal, waste engine oil. The pretreated rice straw was pelletized by using a biomass densification platform, and then the energy consumption and pellet quality were tested. Experimental results indicate that the hydrothermal pretreatment plays an important role in reducing energy consumption and improving product quality, and waste engine oil has a better effect than the rapeseed meal. We also observe that the obtained pellet quality reaches the standard of middle-grade coal, and the proposed pretreatment method realizes the comprehensive utilization of waste agricultural resources.

**Keywords** Rice straw · Pelletization · Hydrothermal pretreatment · Energy consumption · Pellet quality · Optimization

## 1 Introduction

With the growing global energy and environmental problems, clean and renewable energy utilization draws the attention from worldwide [1–3]. Biomass fuel, as one of the most promising renewable energy sources, has become a focus in recent years due to extensive application [4–6]. China is an agricultural country, which has over 20 types of crop straws, and the harvest yield is more than 700 million tons per year. According to the China Statistical Yearbook (2016 Edition),

the yield of rice straw accounted for 33.5 % of the total crops straw yield. However, most of these abundant agricultural biomass resources are abandoned or uncontrolled burned. To eliminate the current energy and environmental problems, it is necessary to make a good use of these biomass resources. Among the various energy utilization ways of crop straw, pelletization is a process which has high productivity, and produces high density and high strength [7–9]. The biomass fuels produced in this way is also convenient for transportation and burning [10,11]. As a result, the biomass pelletization has gradually become the mainstream technology. However, the application of the rice straw pelletization technology has two technical bottlenecks, including high energy consumption and low product calorific value.

To improve the biomass pelletization process, Kong et al. [12] discovered that the product quality could be improved by adding linen fibers. Rosin et al. [13] mixed biomass materials with various additives such as starch, molasses, HSC-residue, slaked lime, ash and montan resin, to improve the product density. Mediavilla et al. [14] found that the physical quality of the biomass pellet was higher when liginosulphonate was added. To increase the calorific value of rye straw pellets, paraffin, palmitin, anthracite coal and lignite coal were mixed in the biomass material [15]. Shang et al. [16] investigated the influence of rapeseed oil on the friction during densification

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**Fig. 1** The briefly map of Jiangyan, Jiangsu province, China

process. Zarringhalam et al. [17] added coal waste into the biomass material and found that the water resistance index and tensile strength of the pellets were improved. Zannikos et al. [18] mixed sawdust with waste plastic to produce pellets and analyzed the calorific value and emissions of the products. Filbakk et al. [19] studied the effect of bark content on the quality of the scots pine pellets. Liu et al. [20] mixed rice straw with bamboo to make pellets.

At present, hydrothermal carbonization is one of the main biomass pretreatment methods [21–26]. Liu et al. [27] carried out experiments and found that hydrothermal treated biomass pellets had higher carbon content and calorific value, and lower ash content, but the compressive strength of the product decreased. Reza et al. [28,29] used hydrothermal pretreated biomass to produce pellet and found that both calorific value and physical quality of the product were improved.

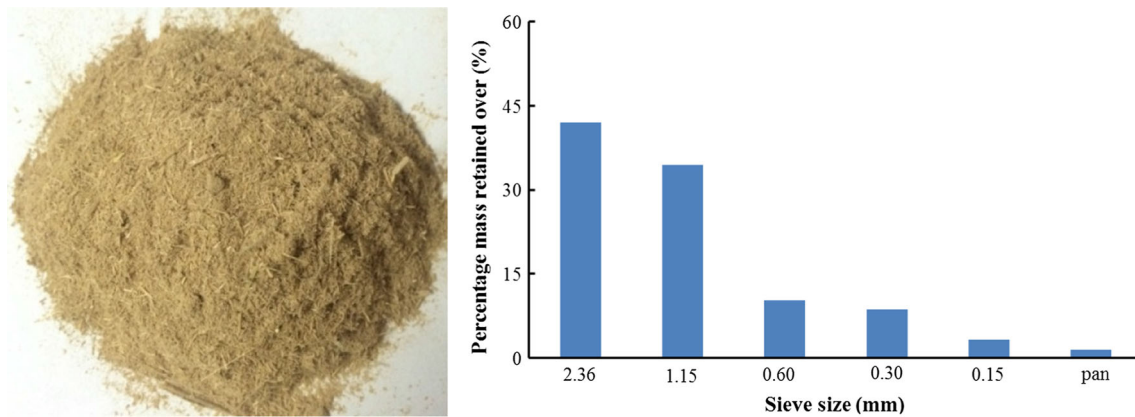
The pretreated biomass material is more homogeneous, which makes it easier to be pelletized and the volumetric energy density could be improved. However, recent researches mainly focused on wood-biomass, and the pretreatment way was usually single, just physical or simple chemical pretreatment. There are few researches related to rice straw material or composite pretreatment. Meanwhile, the material pretreatment had composite influences on energy consumption, die wear and product quality. Therefore, the rice straw was pretreated by a composite pretreatment way which included hydrothermal carbonization treatment and

adding economic additives such as waste engine oil and rape-seed meal firstly in this paper. Then, the pretreated material was pelletized, and energy consumption and pellet quality were tested. Finally, the optimization scheme was selected. The purpose of this research is to reduce energy consumption of the rice straw densification process and increase calorific value of the products, while density and strength of the product meet the relevant standards.

## 2 Experiment and Methods

### 2.1 Materials and Experimental Design

As Jiangsu province is the main rice production region in China, and the planting area of rice in Jiangyan (the brief map is shown in Fig. 1) was over 80 thousand acres, so the selected raw material was rice straw harvested in Jiangyan in the autumn of 2016, and the rice variety is Nangeng 46. 20 kg rice straw was collected in three times from the harvested field, then the sundries were removed, and the sample was dried naturally to the water content of 10–15%. Last, the straw was grinded to 2 mm and stored into ten plastic bags for the following test. The granularity distribution is shown in Fig. 2. Hemicellulose content of the experimental rice straw is 27.2%, cellulose content is 34%, lignin content is 14.2%,



**Fig. 2** Granularity distribution of the grinded rice straw

the stacking density is  $75.3 \text{ kg/m}^3$ , and the calorific value is  $14.753 \text{ MJ/kg}$ .

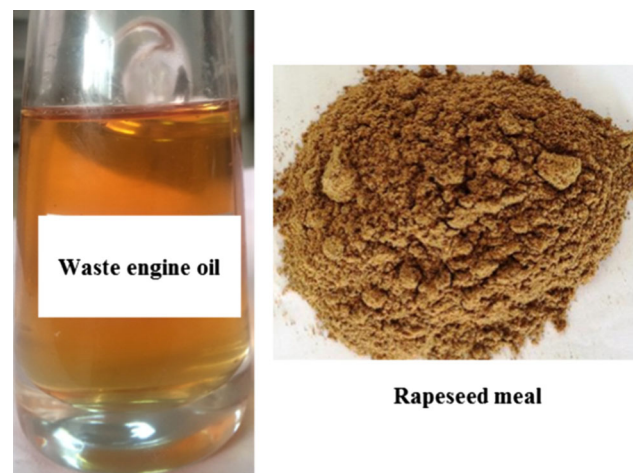
Experiments in this paper mainly focus on the influence of different pretreatment method on energy consumption and product quality of the rice straw pelletization process. Firstly, the non-pretreated rice straw was pelletized. Then, the selected binders were added into the raw materials and the mixtures were pelletized. Finally, the rice straw was hydrothermal pretreated, and the selected binders were mixed into the pretreated rice straw and the mixtures were pelletized. The raw materials were pelletized by a specific pelletter platform which has the function of pelletization, testing energy consumption and tensile strength.

## 2.2 Selection of Additives

The additives should be cheap and could improve the physical and chemical properties of the raw material, increase the calorific value of the product, and reduce the friction during the densification process to reduce energy consumption. Furthermore, the additives should neither have bad influence on the quality of products nor cause environmental pollution. Particularly, the industrial or agricultural waste which need to be treated is the first choice. Therefore, waste engine oil and rapeseed meal are chosen as the additives initially, and the main combustion products of these additives are  $\text{CO}_2$  and  $\text{H}_2\text{O}$ . The additive amount is arranged between 1 and 8%. In addition, the starch was chosen as a general binder and its amount is arranged as 4%.

### 2.2.1 Waste Engine Oil

Lubricating oil is used for lubrication, rust prevention, sealing and cooling in various types of vehicles and mechanical equipment, and the engine oil is a kind of lubricating oil used in engines. Its main components are hydrocarbons, and the calorific value is more than  $30 \text{ MJ/kg}$ . As the engine oil



**Fig. 3** The additives samples (waste engine oil and rapeseed meal)

must be replaced when it is used after a certain period, a large amount of waste engine oil will be produced every year. Making good use of this waste engine oil can not only make waste profitable and save energy, but also protect the environment. The waste engine oil sample is shown in Fig. 3.

### 2.2.2 Rapeseed Meal

Rapeseed is one of the most important oil crops in China, and the annual yield of rapeseed is more than 11 million tons. Rapeseed is mainly used to make rapeseed oil; 350–400 kg oil can be produced from 1 ton rapeseed, and then plenty of rapeseed meal is left over. Although rapeseed meals are rich in protein, they are harmful because of the anti-nutritional ingredients contained in rapeseed meal, such as thioglycoside, erucic acid, phytic acid and polyphenol. This limits its use as protein source for animal feed. Therefore, only a few rapeseed meals could be added into the feed and most of them are directly used as organic fertilizer. In fact, the content of oil, protein and lignin is very high in rapeseed meal,

they will soften and act as excellent lubricants and binders in the pelletization process, and its calorific value is about 19.9 MJ/kg [30]. In this experiment, rapeseed meal is chosen as one kind of additives. The rapeseed meal sample is shown in Fig. 3.

### 2.2.3 Starch

Starch is a renewable carbohydrate which contains a small amount of fat and protein and is widely used as adhesives in biomass densification process [31]. It has been discovered that the quality of the pellets is improved when starch is added into the raw material.

## 2.3 Hydrothermal Pretreatment

Hydrothermal pretreatment is also called auto-hydrolysis. The water is kept subcritically or critically under a high pressure by increasing the temperature. And the stable lignocellulose in biomass materials would be hydrolyzed efficiently in the hydrothermal process. Hydrothermal pretreatment takes water rather than any other chemical reagents as medium. This process is controllable and has short reaction period, which is friendly to the environment. Compared with hydrothermal gasification and hydrothermal liquefaction, hydrothermal carbonization needs lower temperature and pressure, and the reaction condition is moderate. In the hydrothermal carbonization process, the temperature is kept at 180–250 °C and the pressure is about 1.4–27.6 MPa. The products of hydrothermal carbonization are uniform-sized and have high energy density [32].

Biomass hydrothermal carbonization would produce solid, liquid and gaseous products. The solid product is coke, the liquid products include reducing sugar, acetic acid and TOC, and the gaseous product is mainly CO<sub>2</sub>. In this experiment, the rice straw is treated by hydrothermal carbonization, and the solid products are pelletized. Temperature of the pretreatment is 240 °C, and the reaction time is 30 min.

## 2.4 Testing Apparatus and Process

The pelleter device used in this paper is a self-made pelletization platform. This platform is made up of two parts: a pelletization apparatus and an electronic universal testing machine. The pelletization apparatus can simulate the real pelletization environment. A heating element system which can heat the die quickly was adopted in the apparatus. The electronic universal testing machine is produced in HRJ Company in Jinan, Shandong province, China. The maximum test load is 100 kN, the extrusion speed is 0.01–1000 mm/min, the accuracy is  $\pm 0.5$ , the range of load measurement is 0.4–100% FS, and the displacement reso-

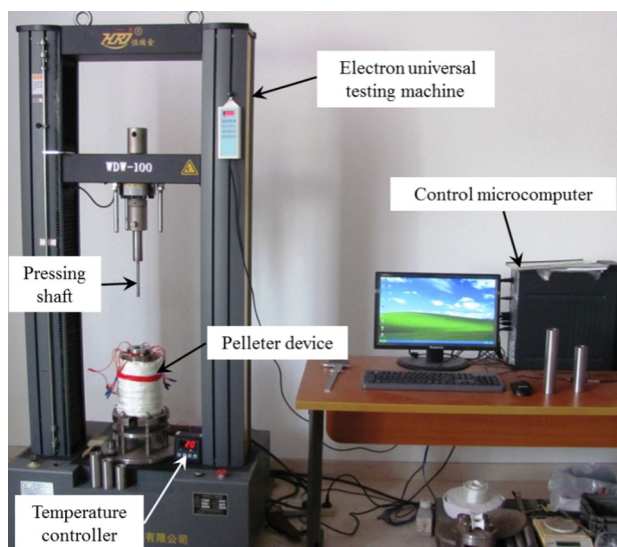


Fig. 4 The pelletization platform

lution is 0.015  $\mu\text{m}$ . The pelletization platform is shown in Fig. 4.

The testing process includes the following, (1) adjusting the pelleter apparatus to make sure the pressing shaft is aligned with the cavity of the die; (2) heating the die to the target temperature, and then adding the weighed material into the die; (3) compressing the materials at a certain speed until the predetermined pressure is reached; and (4) maintaining the pressure for some time and finally extruding the pellet out of the die.

The control computer was used to record the stress, displacement and deformation data during the whole process. In this experiment, the length to diameter ratio of the die is 4, the temperature is 120 °C, the compressing speed is 50 mm/min, the pressure is 60 MPa, and the pressure holding time is 60 s. Moisture content of the pelletization materials is 10%.

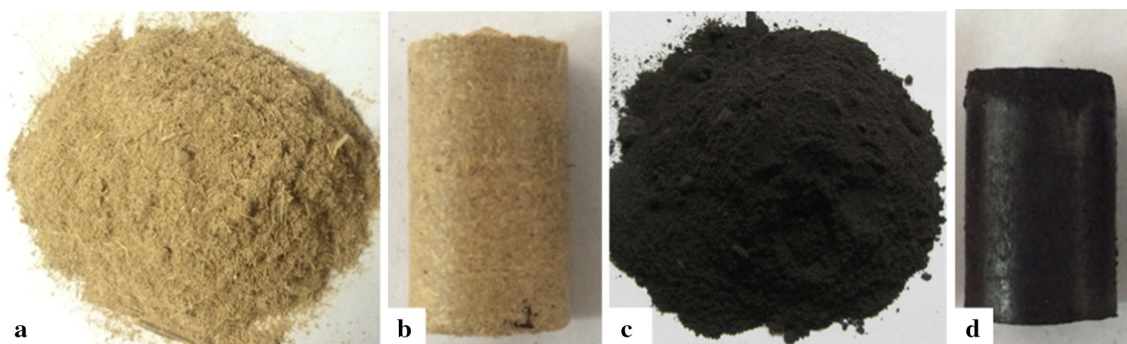
## 2.5 Experiment Testing Indicators

The pelletization platform shown in Fig. 4 was used to compress the rice straw to make pellets. And then several tests were carried out to evaluate the densification process. The testing items include specific energy consumption, tensile strength and pellet density; moreover, calorific value was also obtained through experiment.

### 2.5.1 Specific Energy Consumption

Specific energy consumption means the energy consumption of per unit mass biomass when it was compressed from loose form to densified pellet, which is an important evaluation index of the pelletization process. The displacement–stress





**Fig. 5** Non-pretreated, hydrothermal carbonization rice straw and the corresponding pellet. **a** Non-pretreated rice straw, **b** non-pretreated pellet, **c** hydrothermal carbonization straw, **d** hydrothermal carbonization pellet

curve recorded by the computer can be used to calculate the energy consumption, and then the specific energy consumption can be calculated by formula (1).

$$E = \frac{W}{m} = \frac{S}{m} \int_0^l \sigma(x)dx \tag{1}$$

where  $E$  is the specific energy consumption, J/kg;  $W$  is the total energy consumption of a single pellet, J;  $m$  is the weight of the pellet, g;  $\sigma$  is the pelleting pressure, Pa;  $x$  is the displacement, m;  $l$  is the maximum compressive displacement, m;  $S$  is the cross-sectional area of the pressing shaft, m<sup>2</sup>.

**2.5.2 Tensile Strength**

Tensile strength is used to evaluate the crushing resistance and mechanical stability of the pellets. The pellet with a certain length was placed on the pelletization platform transversely, the pressing shaft pressed it at a certain speed until the pellet was broken, and the maximum compressing force  $F$  was record. The tensile strength is calculated by formula (2).

$$\sigma_t = \frac{F}{dl} \tag{2}$$

where  $\sigma_t$  is the tensile strength, Pa;  $F$  is the compressing maximum force, N;  $d$  is the diameter of the pellet, m;  $l$  is the length of the pellet, m.

**2.5.3 Relaxed Density**

Relaxed density of the pellet is measured based on the standard of NY/T 1881.1-2010 (Densified biofuel-Test methods Part 1: General principle, China). The sample was cooled for 24 h in the air, and then mass of the pellet was measured by an electronic scale, and length and diameter of the pellet were also measured by a vernier caliper. The density of the sample is calculated by formula (3).

$$\rho = m/Vp = \frac{4m}{\pi DL} \tag{3}$$

where  $\rho$  is the relaxed density, g/cm<sup>3</sup>;  $m$  is the mass of the pellet, g;  $D$  is the diameter of the pellet, cm;  $L$  is the length of the pellet, cm.

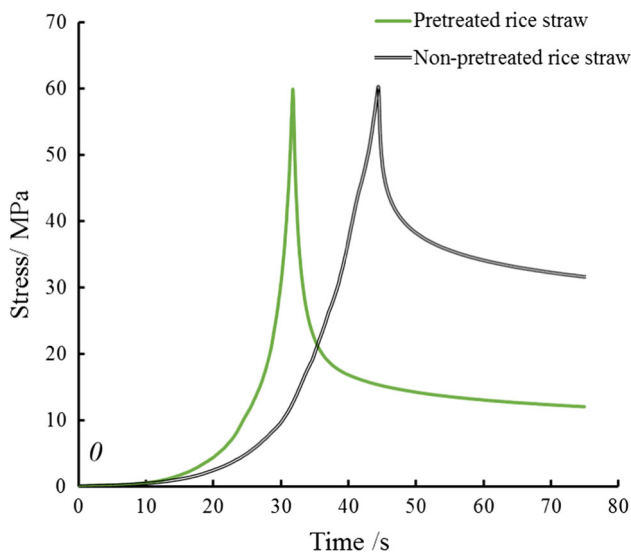
**2.5.4 Calorific Value**

The apparatus used to test the calorific value is an automatic rapid calorimeter, which is made by Shanghai OURUI instruments Equipment co., LTD. The standard relative deviation is less than 0.2 %, and the temperature resolution is 0.001 °C. A weighed pellet was taken as the sample, then a certain length of conductive wire was convolved around the pellet, then the oxygen bomb filled with oxygen was installed into the calorimeter, and the calorific value is measured.

**3 Results and Analysis**

The non-pretreated and pretreated rice straw pellets are shown in Fig. 5. It can be seen from Fig. 5 that the color of pellet becomes darker after hydrothermal carbonization (HTC) treatment. This is mainly due to the increasing content of carbon after hydrothermal carbonization treatment. Figure 6 shows the compaction curves of non-pretreated and pretreated rice straw; specific energy consumption is calculated according the curves. It should be noted that the hydrothermal pretreated straw is difficult to be compressed into pellets without binders.

According to the testing methods introduced in Sect. 2, several parameters including specific energy consumption (SEC), relaxed density (RD), tensile strength (TS) and calorific value (CV) of the products were obtained. The testing results are shown in Table 1.



**Fig. 6** Compaction curves of the non-pretreated and pretreated rice straw

### 3.1 Specific Energy Consumption

In Table 1, it can be found that specific energy consumption of the untreated rice straw is 50.089 J/g and energy consumption of the pretreated rice straw is between 19.492 and 47.392

J/g. It is obvious that the energy consumption decreased when the materials were pretreated. Among all the pretreated methods, the HTC pretreated rice straw with the additives of 10% waste engine oil and 4% starch performed the best; its specific energy consumption was only 19.492 J/g. The influence of each pretreated method on the specific energy consumption is shown in Fig. 7 based on the data shown in Table 1.

Figure 7 shows the influence of different pretreated methods on the specific energy consumption of non-pretreated and pretreated straw. The specific energy consumption gradually decreased with the increase in additive proportion of additives. For the two types of additives, waste engine oil performed better than rapeseed meal in saving energy. And the specific energy consumption of hydrothermal pretreated straw was lower than that of the untreated straw, while the additives were the same.

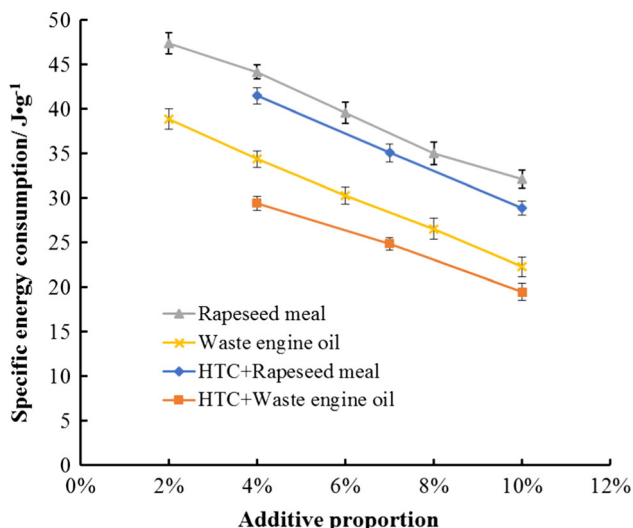
### 3.2 Relaxed Density of the Pellet

Relaxed density of the pellet was measured based on the method in Sect. 2, and the testing results are shown in Table 1. It can be seen from Table 1 that relaxed density of the untreated straw was 1.051 g/cm<sup>3</sup>, while relaxed density of the composite pretreated straw was between 0.975 and 1.268 g/cm<sup>3</sup>. According to the agricultural trade standard

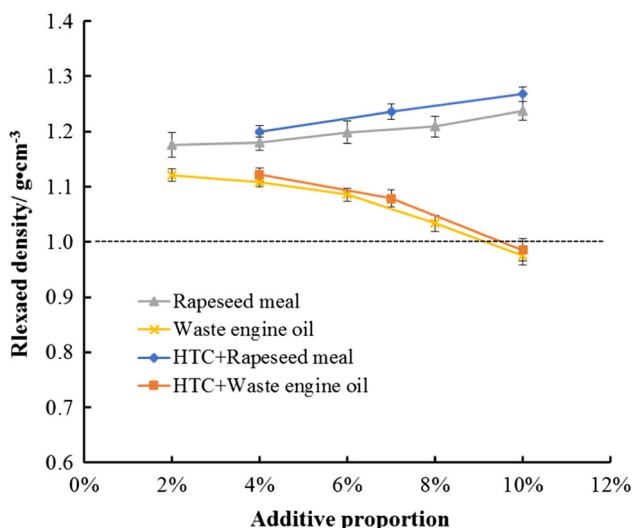
**Table 1** Testing results for the rice straw pellet pretreated in different ways

Test no.	Serial no.	Pretreated method	Additives	Content	SEC (J/g)	RD (g/cm <sup>3</sup> )	TS (MPa)	CV (MJ/kg)
1	D1	None	None	None	50.089	1.051	1.130	14.753
2	G32	None	Rapeseed meal	2%	47.392	1.176	2.103	15.026
3	G33			4%	44.172	1.180	2.351	15.147
4	G34			6%	39.565	1.199	2.526	15.257
5	G35			8%	35.017	1.209	2.734	15.421
6	G36			10%	32.138	1.238	2.806	15.650
7	G42	None	Waste engine oil	2%	38.874	1.121	1.939	16.106
8	G43			4%	34.375	1.108	1.710	16.518
9	G44			6%	30.284	1.086	1.421	16.889
10	G45			8%	26.541	1.033	1.162	17.002
11	G46			10%	22.293	0.975	0.712	17.517
12	SR04D4	Hydrothermal pretreatment	Waste engine oil	4% + 4%D	29.427	1.122	1.045	24.681
13	SR07D4			7% + 4%D	24.857	1.079	0.870	24.918
14	SR10D4			10% + 4%D	19.492	0.985	0.612	25.113
15	SC04D4	Hydrothermal pretreatment	Rapeseed meal	4% + 4%D	41.472	1.200	1.012	20.141
16	SC07D4			7% + 4%D	35.074	1.235	1.231	20.646
17	SC10D4			10% + 4%D	28.879	1.268	1.472	21.173

Note: In Table 1, “D” means the content of starch. For example, “4%D” means the adding mass fraction of starch is 4%



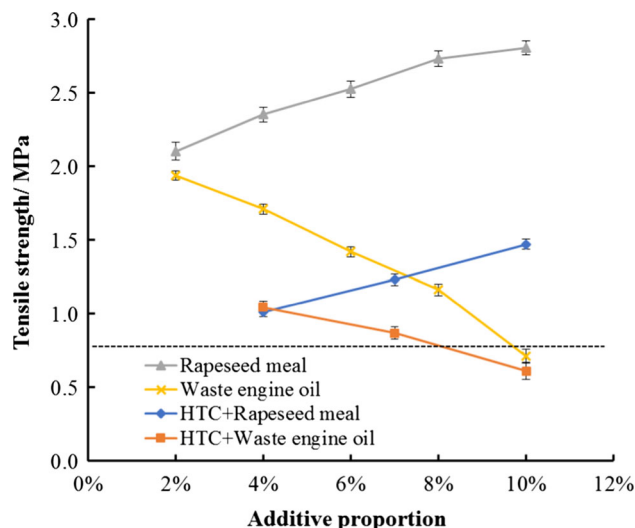
**Fig. 7** The influence of different pretreated method on the specific energy consumption (SEC)



**Fig. 8** The influence of different pretreated method on the relaxed density (RD)

of NY/T 1878-2010 (Specification for Densified Biofuel) in China, density of the herbaceous granular biomass solid fuel should not be less than 1000 kg/m<sup>3</sup>. In other words, the product was qualified only if the density is over 1 g/cm<sup>3</sup>. Based on the obtained data shown in Table 1, the influence of different pretreated ways on density is shown in Fig. 8. The dotted line in Fig. 8 is the density qualification line. If the density is above the dotted line, the products are qualified and the corresponding pretreatment method is feasible. Otherwise, the products are not qualified and the corresponding pretreatment method is infeasible.

Figure 8 shows the influence of different additives on the density of pellet made from untreated materials. When the additive was rapeseed meal, the density increased with the



**Fig. 9** The influence of different pretreated method on the tensile strength (TS)

increase in additive proportion. For waste engine oil, the density decreased with the increase in additive proportion. Rapeseed meal performed better in improving the density. It is observed that relaxed density of the hydrothermal pretreated pellet was improved significantly compared to the untreated straw. The pellet density failed to meet the standard if the additive proportion of waste engine oil was more than 9%. Thus, some of the pretreatment ways could improve pellet density, and some would decrease it.

### 3.3 Tensile Strength of the Pellet

Tensile strength of the pellet was measured based on the method shown in Sect. 2, and the testing results are shown in Table 1. It can be seen from Table 1 that tensile strength of the untreated straw pellet was 1.130 MPa, tensile strength of the pretreated straw was between 0.612 and 2.806 MPa. Lu et al. [33] pointed out that tensile strength of the straw pellet biofuel should not be lower than 0.81 MPa. Thus, 0.81 MPa was taken as the standard of the tensile strength of rice straw pellet in this paper. Based on the obtained data in Table 1, the influence of different pretreated ways on the tensile strength of the pellet is shown in Fig. 9. In Fig. 9, if the tensile strength is above the dotted line, the pellet is qualified and the corresponding pretreated method is feasible. Otherwise, the product is not qualified and the corresponding pretreatment method is infeasible.

Figure 9 shows the influence of different pretreated method on the tensile strength of the pellet. When the additive was rapeseed meal, the tensile strength increased significantly with the increase in additive proportion. Tensile strength of the pellet made from hydrothermal pretreated straw decreased compared to untreated straw. For the waste

**Table 2** Comparison of testing items between the selected pretreated methods

Item no.	Serial no.	SEC (J/g)	Variation range	RD (g/cm <sup>3</sup> )	Variation range	TS (MPa)	Variation range	CV (MJ/kg)	Variation range
1	D1	50.089	–	1.051	–	1.130	–	14.753	–
2	G35	26.541	–47.76%	1.033	–1.71%	1.162	+2.83%	17.002	+15.24%
3	SR07D4	24.857	–51.08%	1.079	+2.66%	0.870	–23.01%	24.918	+68.90%

engine oil, the tensile strength decreased with the increase in additive proportion. Rapeseed meal performed better in improving the tensile strength; when the waste engine oil was more than 8%, tensile strength of the pellet failed to meet the standard. Thus, some of the pretreatment ways could improve the tensile strength, and some would decrease it.

### 3.4 Calorific Value

Calorific value of the pellet was measured based on the method shown in Sect. 2, and the testing results are shown in Table 1. It can be seen from Table 1 that the lower calorific value of the untreated straw pellet is 14.753 MJ/kg and the lower calorific value of the pretreated straw pellet is between 15.026 and 25.113 MJ/kg. We observe that the lower calorific value increased with the increase in the additive proportion of additives. Waste engine oil performed better in improving the lower calorific value. It was concluded that the lower calorific value could improve a lot by hydrothermal pretreatment. For example, when additive proportion of waste engine oil was 4%, the lower calorific was improved by 11.96%. But for the hydrothermal pretreated straw, the lower calorific was improved by 67.29% compared to untreated straw pellet. Therefore, all the pretreated method used in this paper could improve the lower calorific value of the pellet effectively.

### 3.5 Optimal Selection and Analysis

#### 3.5.1 Selection of the Optimal Pretreated Method

The qualified rice straw pellet should have proper density, high tensile strength and calorific value; meanwhile, energy consumption of the densification process should be low. Based on the analysis of the testing results above, two preferable pretreated methods were selected. Specific optimization methods are direct analysis, comparative analysis, and exclude by comparing method. Scheme that consumed the least energy and obtained the highest relaxed density, tensile strength and calorific value pellet was taken as the evaluation criteria, then the unideal test schemes were excluded by comparison and analysis, and the optimal scheme was determined. The selected pretreated methods included untreated rice straw with 8% waste engine oil (G35) addition and hydrothermal pretreated rice straw with 7%

waste engine oil and 4% starch (SR07D4) addition. The selected methods had better comprehensive pellet quality and lower energy consumption. Comparison of testing items between the selected pretreated methods and the untreated rice straw (D1) is listed in Table 2.

We observe from Table 2 that energy consumption of the selected pretreated scheme G35 was 47.76% less than that of the untreated rice straw, and pellet density was decreased by 1.71%, tensile strength was improved by 2.83%; meanwhile, calorific value was improved by 15.24%. However, energy consumption of the selected pretreated scheme SR07D4 was 51.08% less than that of the untreated straw, pellet density was improved by 2.66%, tensile strength was decreased by 23.01%, and calorific value was improved by 68.90%. Therefore, the comprehensive improvement effect of the scheme SR07D4 was the best among the four selected methods.

## 4 Conclusions

In this paper, we pretreated the rice straw by hydrothermal carbonization method and used rapeseed meal and waste engine oil as additives, and then densification energy consumption and pellet quality of the pretreated materials were tested. The following conclusions were drawn:

- (1) Densification energy consumption of the rice straw was gradually decreased with the increase in additive proportion of additives. Waste engine oil performed better than rapeseed meal in saving energy. Among all the pretreated methods, the HTC pretreated rice straw with the additives of 10% waste engine oil and 4% starch performed the best.
- (2) Relaxed density of the rice straw pellet increased with the increase in additive proportion of rapeseed meal, and the density decreased with the increase in additive proportion of waste engine oil. And relaxed density of the pellet made by hydrothermal pretreated straw was improved significantly compared to the untreated straw.
- (3) Tensile strength of the rice straw pellet increased with the increase in additive proportion of rapeseed meal, and decreased with the increase in additive proportion of waste engine oil. Tensile strength of the pellet made from hydrothermal pretreated straw decreased compared to



untreated straw. Some of the pretreatment ways could improve the tensile strength, and some would decrease it.

- (4) Calorific value of the pretreated rice straw pellet increased with the increase in the additive proportion of additives effectively. Calorific value of the pellet improved significantly by hydrothermal pretreatment.
- (5) The rice straw pretreated by hydrothermal carbonization with the addition of 7% waste engine oil and 4% starch has good performance. The energy consumption was 51.08% less than that of the untreated straw, the pellet density was improved by 2.66%, and the calorific value was improved by 68.90%, while the tensile strength was decreased by 23.01% only. Combustion characteristic of this kind of pellet was excellent. We expect that it would be used as a good substitute fuel for coal.

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