

FTIR and Thermogravimetric Analysis of Three Kinds of Nutshells

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Abstract

The main components and pyrolysis characteristics of *Camellia oleifera* Abel shells, *Castanea mollissima* Blume shells, and *Castanea mollissima* Blume shells were analyzed by using FTIR and thermogravimetric methods. The experimental results indicated that the main components of the three kinds of raw materials consisted of cellulose, hemicellulose, and lignin. The highest contents of hemicellulose, cellulose, and lignin were in *Camellia oleifera* Abel shells ($49.34\% \pm 0.07\%$), *Castanea mollissima* Blume shells ($27.34\% \pm 0.01\%$), and *Carya cathayensis* Sarg shells ($49.78\% \pm 0.01\%$), respectively. The pyrolysis processes of three kinds of shells generally included three stages, namely dehydration, pyrolysis, and carbonization. The peak values and the appearance times of their pyrolysis rates were closely related to their compositions.

Keywords

Camellia oleifera Abel Shells, *Castanea mollissima* Blume Shells, *Carya cathayensis* Sarg Shells, FTIR, Thermogravimetric Analysis, Matrix Application

1. Introduction

China is a major producer of *Camellia oleifera* Abel (**Figure 1(a)**), *Castanea mollissima* Blume (**Figure 1(b)**), and hickory *Carya cathayensis* Sarg (**Figure 1(c)**). Shells are the major by-products of these three fruits processing, and the weights of *Castanea mollissima* Blume shells and *Camellia oleifera* Abel shells account for more than 60% of their fresh fruits [1] [2], and the weight of *Carya cathayensis* Sarg shells is also more than 30% of its weight of fresh fruit [3]. According to the data issued by the ministry of agriculture of the People's Republic of China, annual production of *Camellia oleifera* Abel of China is nearly 8 million tons, the production of *Castanea mollissima* Blume is more than 1.7 million

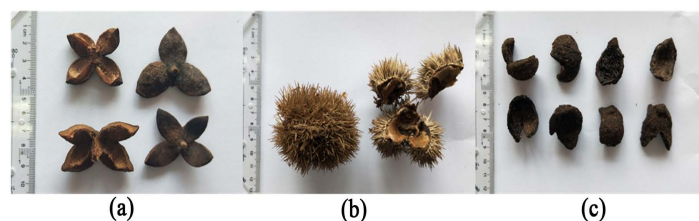


Figure 1. View of three kinds of nutshells.

tons and the production of *Castanea mollissima* Blume is nearly 900 thousands tons in China [4]. Therefore, tons of shells are produced every year. According to the previous studies, in addition to rich in organics, such as polysaccharide, tannin, and saponins, the total contents of cellulose, hemicellulose, and lignin in these shells exceed 80% the weights of their fresh fruits [5] [6], which endows the shells resources with great potentials. However, due to various issues such as difficulty in lignin degradation [7] or incomplete recycle technology, the shells are not utilized efficiently as a resource but treated as waste. These discarded shells are generally stacked or burned, which not only wastes resources but also pollutes the ecological environment [3].

With the prevalence of biomass utilization and the development of new and highly efficient agroforestry concepts, the use of agricultural and forestry waste as the matrix has become a research hotspot. The studied wastes can be pruned branch, fallen leaves, the by-products of fruit processing, and other types of plants residue. The common research topics include the liquefaction and application of liquidized waste (starch substitute in the surface sizing agent in paper-making) [8] [9], industrial production of tar, coke, and emulsion by hot cracking [7] [10], extraction of useful ingredients (tannin, polysaccharide, pigment, etc.) [6] [11], waste compost [12] [13], and manufacture of activated charcoal and biological adsorbents by using waste as raw materials [14]. Indeed, many of the processes have been widely used in the practical applications.

However, there are few studies on composting of *Camellia oleifera* Abel shells, *Castanea mollissima* Blume shells, or *Carya cathayensis* Sarg shells. Composting is a biological and chemical process to convert degradable organic matter into stable humus, which is a kind of continuous and efficient organic waste treatment technology. Moreover, it can effectively alleviate the environmental pressure, enhance soil fertility, and improve soil properties. In the meantime, this process also can alleviate the occurrence of diseases and insect pests to a certain extent [12]. Maturity indices are important standards to estimate the quality of compost products, which means organic matter in the compost is mineralized and finally stabilized. Over-maturity means a large number of nutrients in the waste are wasted, whereas the composting products with less-maturity can cause the decrease of the oxygen content in the soil, in which the harmful substances such as organic acids and H_2S have a negative effect on the normal growth of the plants [15]. Frequently-used maturity indices include temperature, pH, C/N and nitrogen composition, but these indices suffer from some significant limitations,

such as narrow adaptation range, complex detection process, long cycle time, high cost, and challenging in quantification and factory scale composting. Thermal analysis is an effective means to study the thermodynamics and dynamics of various subjects. It can track all kinds of physical and chemical changes in the process of material heating. In particular, thermal analysis is often used to study wood [16]. Bernal [17] used the method of thermogravimetric analysis to analyze the change of compost in the composting experiment of wheat straw as the main raw material and found that the weight loss of the heap at 360°C - 540°C exhibited a good correlation with the maturity, but no specific quantitative index was given. The three kinds of nutshells also contain a large number of wood fibers and polysaccharides. These substances will change significantly during the composting process, and these changes can be detected by the thermogravimetric analysis.

Infrared spectroscopy can identify the characteristic functional groups of the compounds and elucidate the structural changes of the materials. Their measurement needs less sample size, less destructive, simple testing, strong timeliness, and has a good potential for the monitoring of waste compost. In the analysis of municipal solid waste and sludge composting process by infrared spectroscopy, it was found that the variation of spectral characteristics was consistent with the change trend of the heap temperature and the seed germination index GI, which could reflect the change of organic components well and could be used as an index for judging the maturity [18] [19]. However, the conversion of organic components varies under different composting conditions [15].

In this paper, three kinds of shells materials are studied and analyzed by FTIR and thermogravimetric analysis. The composition and pyrolysis characteristics of the fruit shell are determined, which can provide a reference for the further application of three kinds of fruit shells.

2. Experimental Materials and Methods

2.1. Materials

Camellia oleifera Abel shells were obtained from Dongfanghong Forest Farm, Jinhua City, Zhejiang Province, China.

Carya cathayensis Sarg shells (*Carya cathayensis* Sarg) were acquired from Lin'an District, Hangzhou City, Zhejiang Province, China.

Castanea mollissima Blume shells (*Castanea mollissima* BL) were from Qingyuan County, Lishui City, Zhejiang Province.

All the three kinds of shells were collected in October 2017 and separated from fruit just by sheller (shelling by physical means) without any other treatment and exposed under the sunlight for removing excess water.

Then the shells were placed in an oven (DHG-9076A) at 105°C until the weight reached constant. A broken machine (DFT-40) was used to crush the dried shells into powder. Then the powder was sifted through an 80-mesh sieve (GB6003-88) and finally stored in a desiccator as a sample for use.

2.2. Analysis Method

2.2.1. Chemical Composition Analysis

The chemical composition of the shells was determined according to the procedure of Liao [7]. The contents of lignin were determined by method regulated in Chinese National Standard GB/T 2677.8-1994 [20]. The contents of holocellulose were determined with the method of the national standard GB/T 2677.10-1995 [21]. The contents of cellulose were assayed by nitric acid-ethanol method [22]. A method based on GB/T 742-2008 was used to determine the contents of ash [23]. The content of hemicellulose was equal to the difference between the contents of holocellulose and cellulose. Each sample was analyzed three times for calculating the average percentage of the main components.

2.2.2. Fourier Transform Infrared (FT-IR) Spectroscopy Analysis

Fourier Transform Infrared Spectrometer (FTIR, Nicolet iS50, Thermo Fisher Scientific, USA) was used for the identification of various functional groups of the three shells. Oven-dried sample powder 0.001 g (105°C for 1 h) mixed with 0.1 g KBr powder was pressed into the thin and transparent disk by pressed-disk technique and put into FTIR Spectrometer for one test. For each spectrum, a 32-scan absorption interferogram was collected with a resolution of 4 cm⁻¹ in the 400 - 4000 cm⁻¹ region at ambient temperature. Analysis of each sample was repeated three times. The position and peak height of absorption peaks in infrared absorption spectrum were measured by origin 8.0.

2.2.3. Thermogravimetric Analysis

An SDT Q600 (TA Instruments) simultaneous thermal analyzer was used for the analysis of weight loss of the three shells. The experimentation settings were as follows: shells powder samples passed through 80- to 100-mesh sieves, 8 mg in weight, heating rate at 10°C/min, 99.99% nitrogen atmosphere with a flow rate of 50 mL/min, the initial temperature of 30°C, and termination temperature of 800°C [24]. TA analysis and origin 8.0 were used to obtain thermogravimetric curve (TG curve, the relationship between mass and temperature under the programmed temperature) and derivative thermogravimetric curve (DTG curve, obtained by differentially obtaining the thermogravimetric curve and reflected the relationship between the change of sample quality and temperature/time). In the DTG curve, the lowest point on both sides of the absorption peak in the thermogravimetric curve is the cut-off point of the temperature at each stage.

2.2.4. X-Ray Diffraction Analysis

The 80-meshed nutshells powder was dried at the 50°C oven for 6 h and sheeted into thin slices at room temperature, followed by the measurement using a Rigaku D/max 2550 PC type X-ray diffractometer. Experimental conditions were as follows: the X-ray tube was a Cu-target, and the CuK β radiation was eliminated with a nickel sheet with a tube voltage of 40 kV and a tube current of 40 mA. The measurement method was a $2\theta/\theta$ linkage scan. The X-ray crystallinity index was calculated as Equation (1) [25]:

$$C_r I = \frac{I_{002} - I_{am}}{I_{002}} \times 100\% \quad (1)$$

In the formula, $C_r I$ refers to the relative crystallinity (%); I_{002} is (002) lattice diffraction maximum intensity angle; I_{am} represents the scattering intensity of the 2θ angle near the amorphous background diffraction; and I_{am} is the same as the I_{002} unit.

3. Results and Discussion

3.1. Main Components and Contents of Three Kinds of Shells

The contents of cellulose, hemicellulose, and lignin in the three kinds of shells are the material basis for their matrix utilization. The matrix utilization of three kinds of shells mainly depends on the degradation by microorganisms. From the aspect of biodegradation, hemicellulose is the most easily decomposable material, cellulose is a hardly decomposable material, and lignin is an anti-decomposition material [26]. According to methods in 2.2.1, the content of hemicellulose in the *Camellia oleifera* Abel shells was the highest of 49.34%, followed by 33.23% of the *Castanea mollissima* Blume shells, whereas the lignin content in the *Castanea mollissima* Blume shells was the highest of 49.78% (Table 1). From these results, it can be concluded that the *Camellia oleifera* Abel shells and the *Castanea mollissima* Blume shells are relatively optimal substrates for matrix utilization.

3.2. Infrared Spectrum Analysis of Three Kinds of Nutshells

The type and number of chemical bonds can be inferred from the infrared spectral peak assignments of the three shells [27]. As Figure 2 shows, the infrared spectroscopy characterization of absorption peaks was similar among the three shells, for all absorption peaks corresponding to general cellulose, hemicellulose,

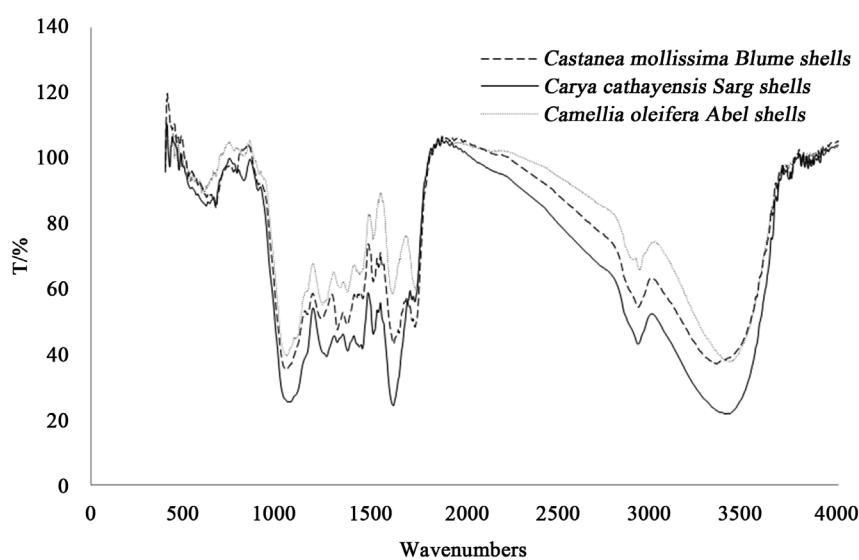


Figure 2. Infrared spectra of three shells.

Table 1. Main ingredient content of three kinds of shells (%).

species	cellulose	hemicellulose	lignin	ash	crystallinity
<i>Camellia oleifera</i> Abel shells	18.62 ± 0.22	49.34 ± 0.07	29.71 ± 0.14	2.57 ± 0.04	39.1
<i>Carya cathayensis</i> Sarg shells	20.63 ± 0.07	22.48 ± 0.02	49.78 ± 0.01	6.88 ± 0.02	64.4
<i>Castanea mollissima</i> Bl. shells	27.34 ± 0.01	33.23 ± 0.02	21.40 ± 0.12	3.22 ± 0.01	46.4

and lignin. The peak at 3420 cm^{-1} corresponded to the hydroxyls of cellulose, hemicellulose, lignin, and water molecules. The absorption peaks at 2927 cm^{-1} , 1452 cm^{-1} , and 1374 cm^{-1} were the characteristic absorption peaks of cellulose, and the peaks at 1615 cm^{-1} , 1508 cm^{-1} , and 1437 cm^{-1} were mainly attributed to the vibration of benzene ring skeleton, which can confirm the existence of lignin. The absorption peak near 1050.46 cm^{-1} was intensive, which corresponds to stretching vibration of C-O bond and alkoxy bond extension in acetyl in cellulose and hemicellulose. The absorption peak near 1734 cm^{-1} caused by the C-O bond of the acetyl group and carboxyl group was a characteristic absorption peak of hemicellulose that is distinguished from other components [12] [16] [28] [29] [30].

It was known from the above spectral analysis that the main components of the three kinds of shells are cellulose, hemicellulose, lignin, and other minor components such as protein and polysaccharide. The location of absorption peaks is related to the type and structure of the substance while the intensity of absorption peak (expressed at the height of the peak) depends on the content of the components in the sample. These components undergo regular changes with the composting process and new substances were produced, which leads to the location and intensity of absorption peaks change as well [29]. Therefore, the location and intensity of absorption peaks can be used as an index for judging the maturity in the composting process [31]. It has been reported that the determination of the content of cellulosic substances by the infrared spectroscopy internal standard method can facilitate the understanding of the changes of the cellulose substances in the composting process and improvement of the composting process, which can probably solve the issue that the thickness of disks significantly affects the intensity of absorption peaks [32] [33]. Moreover, the time required by the infrared spectra analysis is often within two days, which is much shorter than other methods of determining chemical composition, thus tracking the material changes in composting process efficiently and solving the problems in composting process in time.

3.3. Thermogravimetric Analysis

It can be seen from the TG curve in **Figure 3** that the three shells pyrolysis processes exhibited similarities and differences, which are roughly divided into three stages, namely dehydration, rapid weight loss, and slow weight loss stages. The initial temperature of each stage is shown in **Table 2**. In the first stage, the loss of weight mainly included the loss of free water, crystal water, and adsorbed

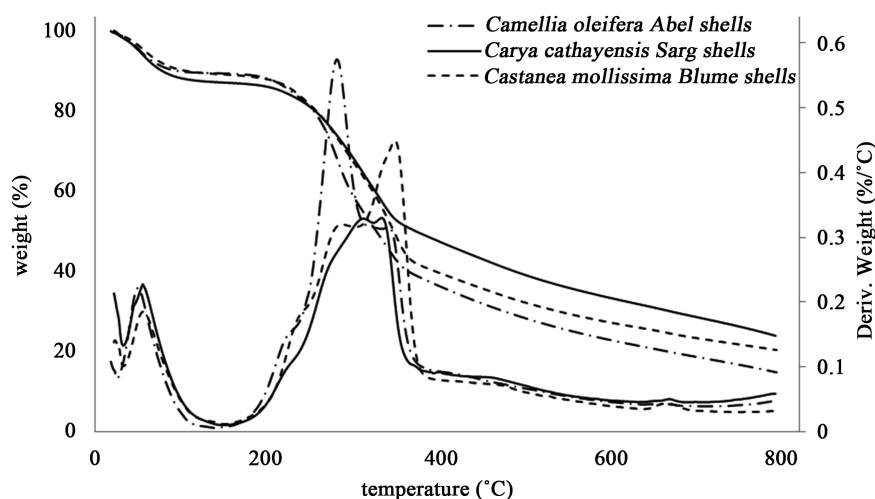


Figure 3. TG curves and DTG curves of three shells.

Table 2. Three-stage initial temperature table for three kinds of shells pyrolysis.

Entry/°C	First stage	Second stage	Third stage
<i>Camellia oleifera</i> Abel shells	25 - 136	142 - 580	>580
<i>Carya cathayensis</i> Sarg shells	33.5 - 150	150 - 395	>394
<i>Castanea mollissima</i> Blume shells	26 - 150	150 - 410	>410

water, as well as the release of small molecules such as terpenes [34] [35]. Dehydration stage of *Camellia oleifera* Abel shells occurred at 25°C - 136°C with weight loss rate of 9.82% and weight loss peak temperature of 50.21°C; this stage of *Carya cathayensis* Sarg shells occurred at 33.5°C - 150°C with a weight loss rate of 10.87% and weight loss peak temperature of 55.87°C; *Carya cathayensis* Sarg shells at 26°C - 150°C with a weight loss rate of 10.28% and weight loss peak temperature of 55.52°C (Table 3). The weight loss wave of the first stage of *Camellia oleifera* Abel shells was 5.31°C - 5.66°C lower than those of the other two shells, which means the shells lost moisture easier. It may be related to the low cellulose content, cellulose crystallinity and the high content of hemicellulose of *Camellia oleifera* Abel shells.

As the temperature continued to rise, the pyrolysis came into the second stage of obvious weight loss. The stage of *Camellia oleifera* Abel shells, *Carya cathayensis* Sarg shells, and *Castanea mollissima* Blume shells respectively occurred between 142°C - 580°C, 150°C - 395°C, and 150°C - 410°C, with weight loss rates of 69.59%, 39.13%, and 50.29%. The weight loss peak temperatures of *Camellia oleifera* Abel shells, *Carya cathayensis* Sarg shells, and *Castanea mollissima* Blume shells were at 282°C and 341.81°C, 313.07°C and 334.16°C, and 287.02°C and 348°C, respectively (Table 3). At this pyrolysis stage, most of the hemicellulose, part of cellulose and lignin take place pyrolysis reactions, and volatile materials are generated that are then carried away by the nitrogen gas flow, which leads to a rapid decline in the TG curve [36]. At this stage, some glycosidic

Table 3. Pyrolysis peak temperatures for three kinds of shells.

Entry/°C	<i>C. oleifera</i> Abel shells	<i>C. cathayensis</i> Sarg shells	<i>C. mollissima</i> Blume shells
Weight loss peak in dehydration stage	50.21	55.87	55.52
Left pyrolysis weight loss peak	282.00	313.07	287.02
Maximum weight loss peak temperature	341.81	334.16	348.00

Remark: *C. oleifera* Abel shells (*Camellia oleifera* Abel shells); *C. cathayensis* Sarg shells (*Carya cathayensis* Sarg shells); *C. mollissima* Blume shells (*Castanea mollissima* Blume shells).

bonds of hemicellulose, cellulose, and lignin in the shells start to break. Simultaneously, some C-O and C-C bonds start to break and produce new products and low molecular weight volatile compounds [37] [38]. Since the pyrolysis of hemicellulose occurs mainly at 200°C - 300°C [39], the weight loss peaks on the left side of the weight loss wave are primarily attributed to the pyrolysis of polymers led by hemicellulose [40] [41], which is consistent with result that the weight loss of the three shells is directly proportional to the hemicellulose content. Since cellulose exhibited an inconspicuous peak at 330°C in TG analysis [42] the right side of weight loss peak is mainly caused by the pyrolysis and volatilization of cellulose-led polymers in the raw material [7] [25]. Lignin exhibited an inconspicuous peak at 350°C - 420°C in TG analysis [41]. The degradation temperature of lignin was between 200°C and 500°C, falling into the entire main pyrolysis stage [43] [44]. In the third stage, the TG curve varied slowly, and the weight loss was relatively slow, which is primarily due to the further degradation of some residual lignin and charcoal [45] [46].

Since the contents of the three major components of the shells are different, their shapes, temperatures, and numbers of weight loss peak vary quite significantly in the second stage. It can be seen from **Figure 3** that the left side of weight loss peak of *Camellia oleifera* Abel shells exhibited an independent apex, whereas the right peak was not obvious. The left peak was significantly higher than the right weight loss peaks of other two raw materials. *Castanea mollissima* Blume shells showed a sharp and inconspicuous peak on the right side while the left side peak was not obvious. The weight loss peak on the right side was significantly higher than that on the left side, which also higher than the right peaks of the other two shells. The weight loss peaks on both left and right sides of the *Carya cathayensis* Sarg shells during this stage were not obvious. The peak temperature corresponding to *Camellia oleifera* Abel shells pyrolysis was the lowest, while the peak temperature on the weight loss of *Carya cathayensis* Sarg shells pyrolysis was the highest. After 400°C, the weight loss trend of all raw materials tended to slow, which is the carbonization stage. At this stage, the pyrolysis of hemicellulose and cellulose is almost finished, and the pyrolysis of the C=C bonds between the benzene ring structures is also basically completed, as well as the side chain of the benzene ring structure in the lignin molecule. However,

since the benzene ring structure of the lignin molecule is relatively hard to decompose, pyrolysis of it forms more fixed carbon. Therefore, the TG and DTG curves tend to be flat [15].

Table 1 and **Figure 2** show that the content of hemicellulose in *Camellia oleifera* Abel shells was 49.34%, higher than the content combined of cellulose and lignin. The content of cellulose only accounts for 18.62%, which is significantly lower than that of hemicellulose. Therefore, the left peak caused by the pyrolysis of hemicellulose and lignin obviously exists, but the right weight loss peak caused by the co-pyrolysis of cellulose and lignin is not obvious and is slightly shoulder-shaped. The content of hemicellulose in the *Castanea mollissima* Blume shells was 33.23%, and the content of cellulose was 27.34% that was the highest among these three shells with a cellulose crystallinity of 46.4%. As a result, the left side peak was not obvious, whereas the right side of weight loss peak caused by the pyrolysis of cellulose and lignin clearly exists. The lignin content of *Castanea mollissima* Blume shells was as high as 49.78%, which is much higher than the lignin contents of the other two raw materials. The cellulose crystallinity was also the highest of 64.4% among the three raw materials while the hemicellulose content was only 22.48%. Therefore, the left peak caused by the pyrolysis of hemicellulose is inconspicuous, and the temperature at which the peak appears is significantly higher than the temperatures at the left peaks of the other two raw materials. In the meantime, due to the high degree of crystallinity of cellulose, the right peak caused by the degradation of cellulose and lignin is also not obvious [30]. Therefore, it can be concluded that the different materials have different weight loss rates and weight loss peaks because of different physical structures and chemical compositions. For a chemical composition, the higher its content, the earlier its weight loss peak appears and the longer the duration of the peak. Since the three TG-DTG curves of the three shells can well reflect different contents and components of the three shells, the curves can also reflect the changes of components in the shells [47]. With the changes of raw material in composting process, the onset and duration of weight loss peaks will also change. These changes can reflect the degree of the composting. By using the thermogravimetric analysis, the weight loss rate and the time of the weight loss peak can be used as one of the indices to judge the degree of maturity. It may also be possible to identify a period of slow degradation during composting, which will facilitate the improvement of the composting process and quality.

4. Conclusions

Up to now, there is no clear standard for judgment of compost maturity. Infrared spectroscopy and thermogravimetry are easy to operate, with strong timeliness and accuracy, and they can directly reflect the changes of substance in composting. Sun [29] and Bernal [17] have mentioned these two methods, but due to the complexity of composting materials and conditions, these two methods do not have quantitative indicators. In the article, three kinds of shells were analysed

by infrared spectroscopy and thermogravimetry for verifying the practicability and feasibility of the two methods. The following conclusions can be drawn from the experiment:

1) From the infrared spectra of the three kinds of shells, it can be concluded that the three main components of the shells are cellulose, hemicellulose, and lignin. There are differences in the content of each component. The content of cellulose from high to low is *Castanea mollissima* Blume shells, *Carya cathayensis* Sarg shells, and *Camellia oleifera* Abel shells. The highest content of hemicellulose in the three shells is *Camellia oleifera* Abel shells, followed by *Castanea mollissima* Blume shells, and *Carya cathayensis* Sarg shells. The content of lignin is in the order of *Carya cathayensis* Sarg shells, *Camellia oleifera* Abel shells, and *Castanea mollissima* Blume shells. The variation in the FTIR of the three components during the composting process can reflect the change of material components and content in each stage, which can help to understand the specific change process of the material during the composting process and solve the problems in the process of composting quickly and effectively.

2) The pyrolysis process of three kinds of shells consists of three stages, namely dehydration, pyrolysis, and carbonization stages. The first stage is the loss of crystal water in the temperature range of 25°C - 150°C with a maximum water loss rate at about 55°C. The second stage is the pyrolysis process of woody fiber, which is the main component of the shells. The starting temperature of hemicellulose pyrolysis is the lowest with maximum weight loss peak at 290°C - 310°C. The pyrolysis temperatures of cellulose and lignin are relatively high, whose maximum weight loss peak at around 340°C. Due to the difference in the contents of the three major components, there are differences in the temperature and shape of the weight loss peaks. The weight loss peak temperature of the three kinds of shells is proportional to the content of each component and cellulose crystallinity. Lower content and higher crystallinity make the peak appear at a higher temperature. And the residual carbon ratio of the *Carya cathayensis* Sarg shells was the highest (23.98%), followed by the *Carya cathayensis* Sarg shells (20.33%), and the lowest residual carbon content was the *Camellia oleifera* Abel shells (14.86%). The lignin content of the *Carya cathayensis* Sarg shells was the highest among the three kinds of shells, and the content of *Camellia oleifera* Abel shells was the lowest. By the results of this study, the results of thermogravimetric analysis of different components have a direct visual difference, which directly reflects the difference in the material content, and the process of compost is also a process of material change. Thermogravimetric analysis can be used to identify the change of the material content during the composting process.

3) Thermogravimetry can reflect the change of material content in the process of composting, while infrared spectroscopy can be used for detection accurately. So TG-FTIR can be used to accurately determine the change of materials in the process of composting.

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Conflicts of Interest

The authors declare no conflicts of interest regarding the publication of this paper.

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