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Original Research Article

Effect of reaction temperature and raw material particle size on the composition of corn straw pyrolysis biological oil

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ABSTRACT

Quartz sand was used as bed material in a small fluidized bed reactor with 1 kg/h feed. Corn straw powder with particle size of 20–40 mesh, 40–60 mesh, 60–80 mesh and 80–120 mesh was used as raw material for rapid pyrolysis at reaction temperatures of 400 ℃, 450 ℃, 500 ℃ and 550 ℃. The bio-oil obtained after liquefaction of pyrolysis gas was analyzed. The variation trend of bio-oil composition in pyrolysis of corn straw powder with different reaction temperatures and raw material sizes was compared. The results show that: (1) the content of 3-hydroxyl-2-phenyl-2-acrylic acid in bio-oil increases with the decrease of raw material particle size, but it is less at 450 ℃; (2) with the increase of reaction temperature, the content of hydroxyacetaldehyde in bio-oil increases at first and then decreases: the content of hydroxyacetaldehyde in bio-oil is the highest at 500 ℃ when the particle size is 20–40 mesh, and the highest at 450 ℃ with the other three particle sizes. Compared with other particle sizes, raw material with the particle size of 60–80 mesh is not conducive to the formation of aldehyde compounds; (3) the reaction temperature of 500 ℃ and the particle size of 60–80 mesh of raw materials are more conducive to the formation of phenolic compounds in bio-oil; (4) the ester compounds with particle size of 20–40 mesh in bio-oil is 20% higher than that of other particle sizes; (5) the reaction temperature and the particle size of raw materials had no significant effect on the formation of ketones, alcohols and alkane compounds in bio-oils.

Keywords: Corn Straw Powder; Fluidized Bed; Reaction Temperature; Raw Material Particle Size; Bio-Oil Composition

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1. Introduction

With the development of society, energy and environment problems have become the focus of attention in today's world. At present, countries all over the world are committed to the development of efficient and pollution-free biomass energy utilization technology to protect their own mineral energy resources and reduce the pollution caused by fossil energy consumption to the environment[1]. Biomass energy can be efficiently exploited through biomass energy conversion technology[2,3]. Biomass energy produces a variety of clean energy and chemical products, thus reducing human dependence on fossil energy. The rapid pyrolysis of biomass is to put biomass raw materials in the pyrolysis unit. By strictly controlling the heating rate and reaction temperature, it is rapidly heated to a higher temperature under the condition of complete anoxia or oxygen deficiency, which leads to the decomposition of macromolecules and the production of non-condensable gases and condensable volatile matter, as well as a small amount of coke product[4,5]. The bio-oil obtained by rapid pyrolysis has shortcomings of large moisture content, high oxygen content, high viscosity, strong acidity, low calorific value and instability, which limit the application of bio-oil as a high-grade liquid fuel[6-8]. Some studies have shown that the composition and content of some macromolecular weight organic compounds in biological oil have a definite effect on the characteristics of biological oil, while the raw material composition and pyrolysis liquefaction reaction conditions will affect the formation of some macromolecular weight organic compounds, especially the reaction temperature has a great influence on the composition of biological oil. For example, the analysis of sawdust pyrolysis biooil by Horne *et al.*[10] of the University of Melbourne found that when the reaction temperature was 400–550 ℃, the main component of bio-oil was oxygen-containing polar compound, which had high oxygen content but low viscosity. Wang *et al.*[8] of Zhejiang University used seven kinds of biomass such as Pinus camphora, pear wood, bamboo powder, rice husk, rice straw, elephant grass and seaweed for pyrolysis. It was found that there were definite differences in the mass fraction and representative compounds of the main components and representative compounds obtained by pyrolysis of different kinds of biomass, and the quality fluctuated in the specific types of compounds, and the phenols in the pyrolysis bio-oil of Pinus camphora were obviously enriched. In addition, Bridgeman *et al.*[11] analyzed the pyrolysis bio-oil of switchgrass and Phalaris arundinacea with particle size less than 90 μm and between 90–600 μm. The bio-oil produced by biomass with small particle size had higher inorganic content, and the particle size decreased the formation of levoglucosan from cellulose decomposition, but the impurity particles in bio-oil increased. By understanding the effect of bio-oil production conditions on bio-oil composition, mastering the relationship between bio-oil characteristics and pyrolysis reaction conditions, in the actual production process, we can change pyrolysis conditions, get different components of bio-oil, and bio-oil can be used as raw materials for separation or transformation to obtain high value-added chemicals, so as to achieve refining of oil products, optimizing the quality of bio-oil. Thus the effective utilization of bio-oil can be improved[12]. However, at present, there is no mature pyrolysis process conditions data to achieve targeted regulation of bio-oil components. How to control the reaction conditions so that the obtained bio-oil characteristics meet some specific requirements, i.e., the realization of targeted regulation is of great significance to the utilization of bio-oil.

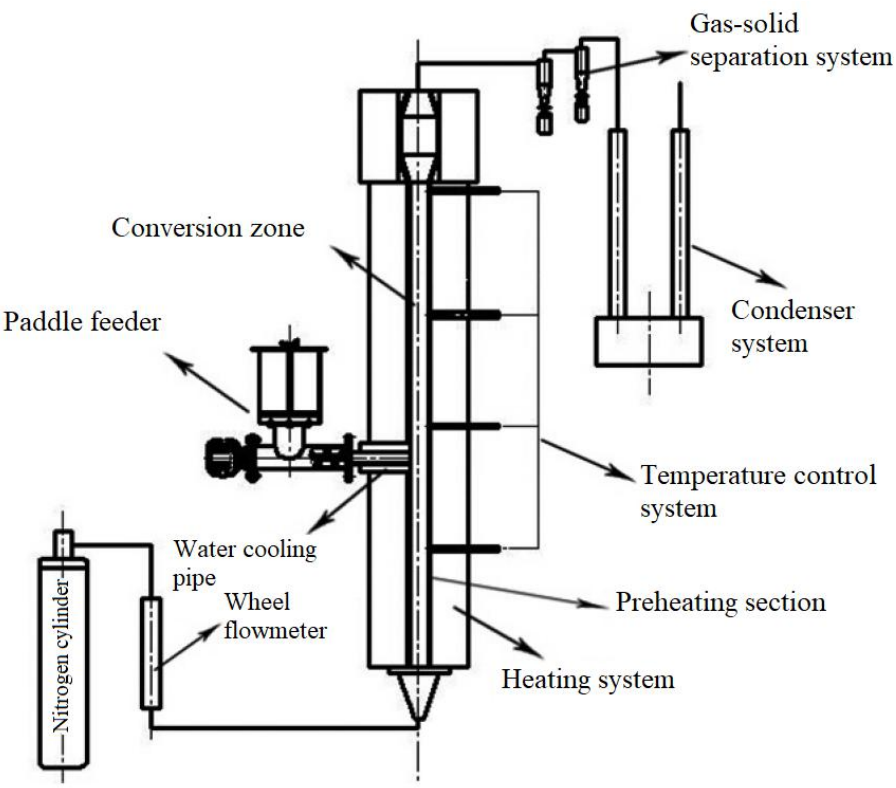

Figure 1. Fluidized bed for fast pyrolysis of biomass.

2. Experiment and test

2.1 Test equipment

In this experiment, a small fluidized bed biomass rapid pyrolysis test device with feed quantity of 1 kg/h was selected, which is mainly composed of dialing feeder, fluidized bed reactor, heating system, gas-solid separation system, condensation system and product collection system. The device uses nitrogen as hot carrier gas, quartz sand as bed material. The degree of reactor temperature is mainly provided by the heating ring wrapped in the outer layer of the reactor, and four K-type thermocouples are evenly distributed to monitor the temperature in the reactor[13]. The rapid pyrolysis of biomass in a small fluidized bed is shown in **Figure 1**.

2.2 Test raw materials

In this experiment, corn straw powder was selected as raw material. The corn straw was harvested near Zhangdian District, Zibo City, Shandong Province in September 2013. After drying for a certain period of time, the corn straw powder was crushed by FC-300 grinder in the laboratory, and the particles were large. In order to obtain the corn straw powder with small particle size, the corn straw powder with the required particle size range was obtained by using JSP-100 high speed multifunctional grinder to continue to crush and screen with sample separation screen. Before each test, the raw materials were placed in the 101-0 electric blast drying box, dried continuously at 105 ℃ for 24 hours, and the moisture content was less than 10%. The industrial analysis data and element analysis data of corn straw powder are shown in **Table 1**.

Table 1. Industrial analysis and elemental analysis of corn stalk

Industrial analysis/%			**Elemental analysis/%**				
Volatile matter	Fixed carbon	Ash content	[C]	[H]	[O]	[N]	[S]
74.92	18.89	5.72	44.22	6.13	42.69	0.18	0.02

2.3 Test conditions

The rapid pyrolysis process of biomass is affected by many factors, such as material type, raw material particle size, reaction temperature, feeding rate and carrier gas flow rate[6,14,15]. The reaction temperature and raw material particle size are the variable factors investigated in this experiment, so it is necessary to control other factors to make them remain unchanged. Four kinds of corn straw powder with particle size of 20–40 mesh, 60–80 mesh and 80–120 mesh were used as raw materials for rapid pyrolysis at temperatures of 400 ℃, 450 ℃, 500 ℃ and 550 ℃ respectively. Quartz sand with particle size of 20–60 mesh was used as bed material, and the feeding rate of feeder was controlled to 1 kg/h, and the nitrogen flow rate was adjusted to 6 m^3/h.

2.4 Test process

A total of 16 kinds of biooils were obtained under different conditions. The instrument used for the composition analysis of the biooils was produced by Agilent of the United States, which is the 6890/5973N type gas chromatography-mass spectrometry, i.e. GC-MS. In order to remove the tiny particle impurities in the bio-oil, the bio-oil obtained under different conditions was filtered many times before the composition detection was carried out. In order to better separate and detect the main components in bio-oil, the analytical conditions of GC-MS should be set as follows[16]:

GC: the inlet temperature is 280 ℃; the shunt ratio is 80:1; the carrier gas is Helium; the DB-1701 capillary column (60 m × 0.25 mm × 0.25 μm); the column temperature is programmed: the initial temperature is 60 ℃, the heating rate is 10 ℃/min till the temperature reaches 240 ℃, and it is kept for 10 min.

MS: electron impact ion source; electron energy is 70 ev, scanning range is 12–550 amu, ion source temperature is 150 ℃, interface temperature is 240 ℃.

3. Test results and discussion

3.1 The effect of reaction temperature on the composition of corn straw pyrolysis bio-oil

The obtained 16 kinds of bio-oil were analyzed with GC-MS. The results showed that the composition of biological oil was complex, including esters, acids, phenols, ketones, alcohols and aldehydes, as well as a small amount of ether and alkane and other compounds. Taking the bio-oil with a particle size of 60–80 mesh as an example, the GC-MS analysis results are shown in **Figure 2**, and **Table 2** shows the comparison of the main components of the four kinds of bio-oil.

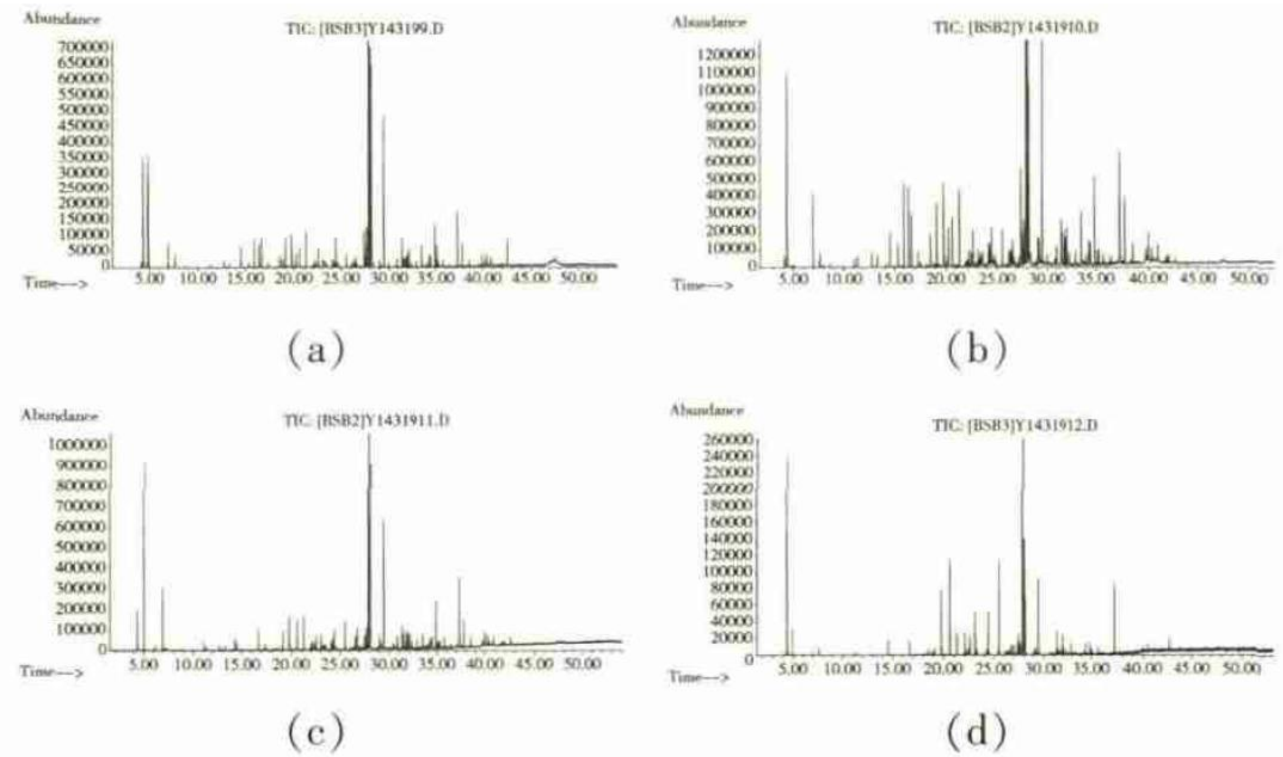

Figure 2. Total ion current diagram of corn stalk bio-oil. **(a)** reaction temperature 400 ℃; **(b)** reaction temperature 450 ℃; **(c)** reaction temperature 500 ℃; **(d)** reaction temperature 550 ℃.

It can be seen from **Table 2** that the content of 3-hydroxyl-2-phenyl-2-acrylic acid in bio-oil is the lowest at 450 ℃ and the highest at 550 ℃, which indicates that the reaction temperature plays a major role in the formation of 3-hydroxyl-2-phenyl-2-acrylic acid. **Figure 3(a)** is the changing trend of the content of 3-hydroxyl-2-phenyl-2-acrylic acid in 16 kinds of bio-oil. It can be seen that the content of 3-hydroxyl-2-phenyl-2-acrylic acid in the same particle size decreases at first and then increases with the increase of reaction temperature, and the content reaches its highest level when the temperature is 550 ℃. It can be seen that the pyrolysis temperature of 450 ℃ is not conducive to the formation of 3-hydroxyl-2-phenyl-2-acrylic acid.

The content of hydroxyacetaldehyde in biological oil is the highest, and the change trend of hydroxyacetaldehyde content with reaction temperature is shown in **Figure 3(b)**. With the increase of temperature, the content of hydroxyacetaldehyde increased at first and then decreased, but the effect of reaction temperature on the content of hydroxyacetaldehyde was also different with different particle size: when the particle size was 20–40 mesh, the content of hydroxyacetaldehyde was the highest at 500 ℃, while the content of hydroxyacetaldehyde was the highest at 450 ℃ when the particle size is the other three kinds.

For alcohol compounds, cyclopropyl methanol only exists in the bio-oil at 500 ℃, and the content is about 3.5%. There is no cyclopropyl methanol detected in the bio-oil at other reaction temperatures. In addition, the content of cyclopentyl alcohol at 550 ℃ was significantly higher than that at other reaction temperatures, as shown in **Figure 3(c)**.

In order to compare the contents of pyrolysis biooil components at different reaction temperatures, the bio-oil components with different particle sizes were counted, and the results were shown in **Figure 4**. Taking the bio-oil with particle size of 60–80 mesh as an example, it can be seen from **Figure 4** that the content of acid compounds is the highest, and it tends to decrease with the increase of reaction temperature, but the overall content does not change much.

Table 2. The main components comparison of corn stalk bio-oil at the four reaction temperatures

Compound name	**Peak area/%**			
	400 ℃	**450 ℃**	**500 ℃**	**550 ℃**
Hydroxyacetaldehyde	4.15	6.18	4.15	2.43
Acetic acid	0.93	0.89	0.85	0.68
1.1-diethyloxyethane	0.76	-	-	0.60
Hydroxyacetone	-	0.92	-	-
Glycol	-	1.00	1.22	1.15
Methyl acetate	-	0.97	0.91	0.83
Butyraldehyde	2.06	2.19	1.57	-
2-furfuryl alcohol	0.83	0.84	-	0.82
2.5-diethyloxytetrahydrofuran	1.72	1.72	1.63	-
2-hydroxyl-2-cyclopentenone	1.34	1.53	1.64	0.94
2(5H)-furanone	0.86	0.95	0.98	0.78
2.2-diethyl-3-methyl oxazolidine	1.20	1.69	-	-
3-methyl-2-hydroxyl-2-cyclopentenone	2.00	2.70	2.82	2.84
1.1-diethyloxypentane	0.78	1.08	-	0.61
Phenol	0.73	1.04	1.39	2.99
4-methylphenol	-	-	1.13	1.80
3-ethyl-2-hydroxyl-2-cyclopentenone	-	0.70	1.50	0.60
5-butyl nonane	-	-	-	1.88
2-methylundecanal	-	-	1.94	-
3-methyl-2.4-furan diketone	-	-	1.92	1.34
2-methoxyphenol	1.23	1.54	1.57	0.92
Cyclopropyl methanol	-	-	3.54	-
4-ethyl phenol	-	0.62	1.02	2.97
5-hydroxyl-2-furan formaldehyde	-	0.71	0.95	-
Cyclopentanol	1.47	1.45	1.06	3.31
Diethyl succinate	1.35	2.13	-	0.09
Glucose	0.72	1.68	0.94	1.53
3-hydroxyl-2-phenyl-2-acrylic acid	16.78	14.62	18.74	27.20

2-methoxy-4- vinylphenol	6.15	6.29	7.10	3.36
Propyl butyrate	7.89	4.47	-	3.10
Hydroquinone	-	0.60	0.88	1.00
2.6-dimethoxyphenol	4.54	4.96	5.41	2.98
4-methoxy-3- methylphenol	0.73	0.92	0.90	-
3-hydroxyl-4- methoxybenzaldehyde	0.67	-	-	0.52
Lauric acid-2.3- dipropyl ester	1.99	2.79	-	-
Octadecaenoic acid methyl ester	1.21	1.19	1.50	-
3-tert butyl-4-hydroxyanisole	-	-	2.40	-
Diethyl phthalate	0.79	0.67	-	0.87
Levoglucosan	2.36	3.09	4.01	3.08
2.6-dimethoxy-4-propene phenol	0.99	1.19	1.16	-

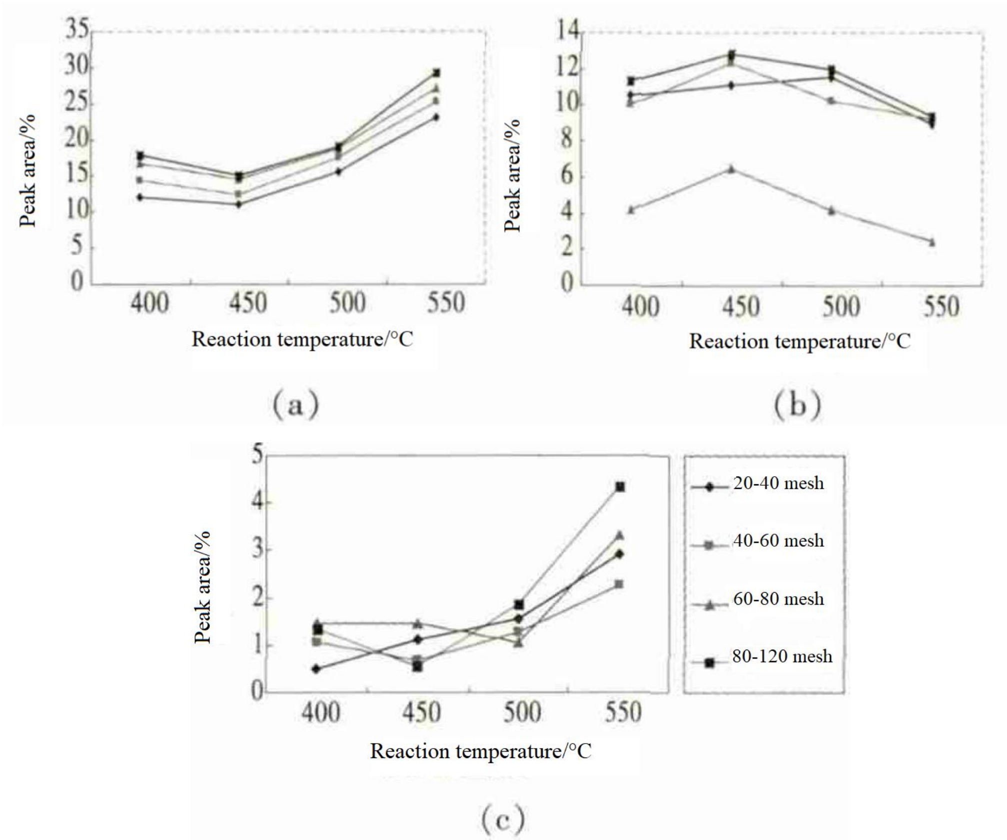

Figure 3. Relationship of bio-oil composition and reaction temperature. **(a)** 3-hydroxy-2-phenyl-2-acrylic acid; **(b)** hydroxyl alcohol; **(c)** cyclopropyl carbinol.

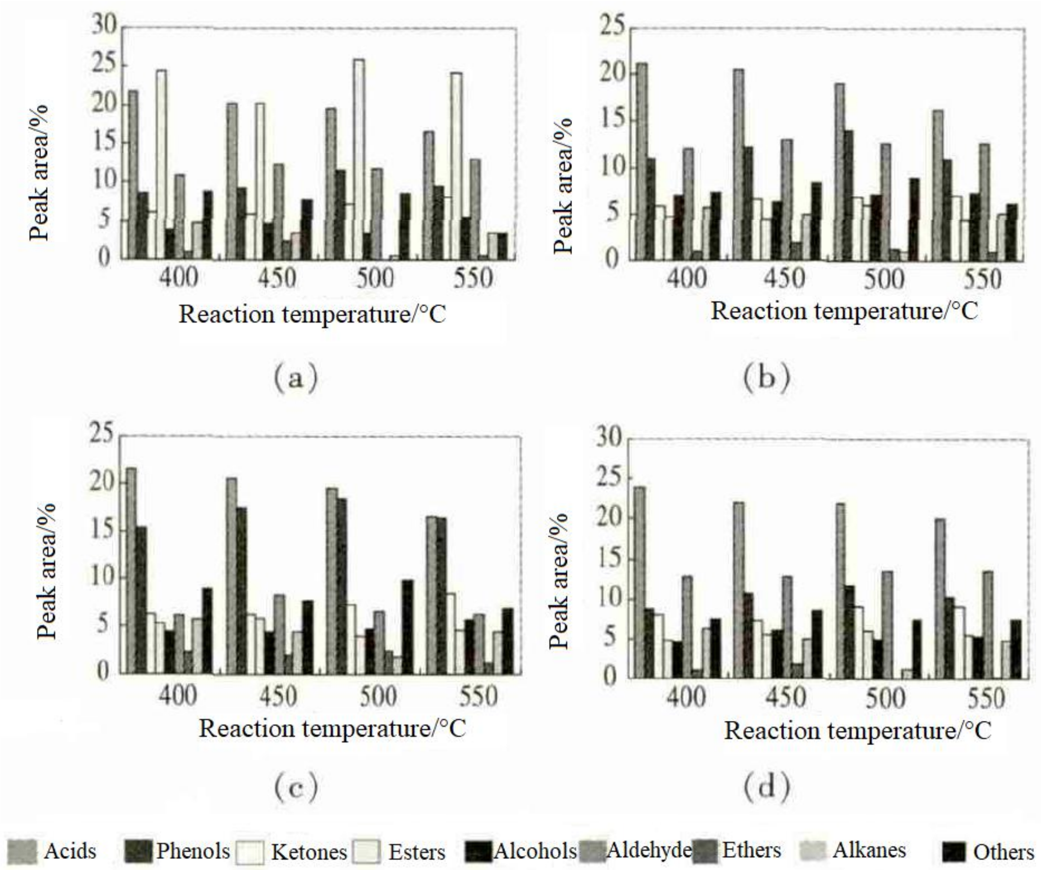

Figure 4. Distribution of the main chemical families of bio-oil. **(a)** particle size 20–40 mesh; **(b)** particle size 40–60 mesh; **(c)** particle size 60–80 mesh; **(d)** particle size 80–120 mesh.

When the particle size of the same raw material is the same, the content of phenolic compounds increases at first and then decreases with the increase of reaction temperature, and the content is the highest at 500 ℃, but the overall content does not change much, which indicates that the reaction temperature of 500 ℃ is more beneficial to the formation of phenolic compounds in bio-oil than the other three reaction temperatures. The contents of ketones, alcohols, ethers and alkane compounds in bio-oils are relatively small, and it does not fluctuate with the change of reaction temperature, which indicates that the reaction temperature has no significant effect on the formation of ketones, alcohols, ethers and alkane compounds in the bio-oil.

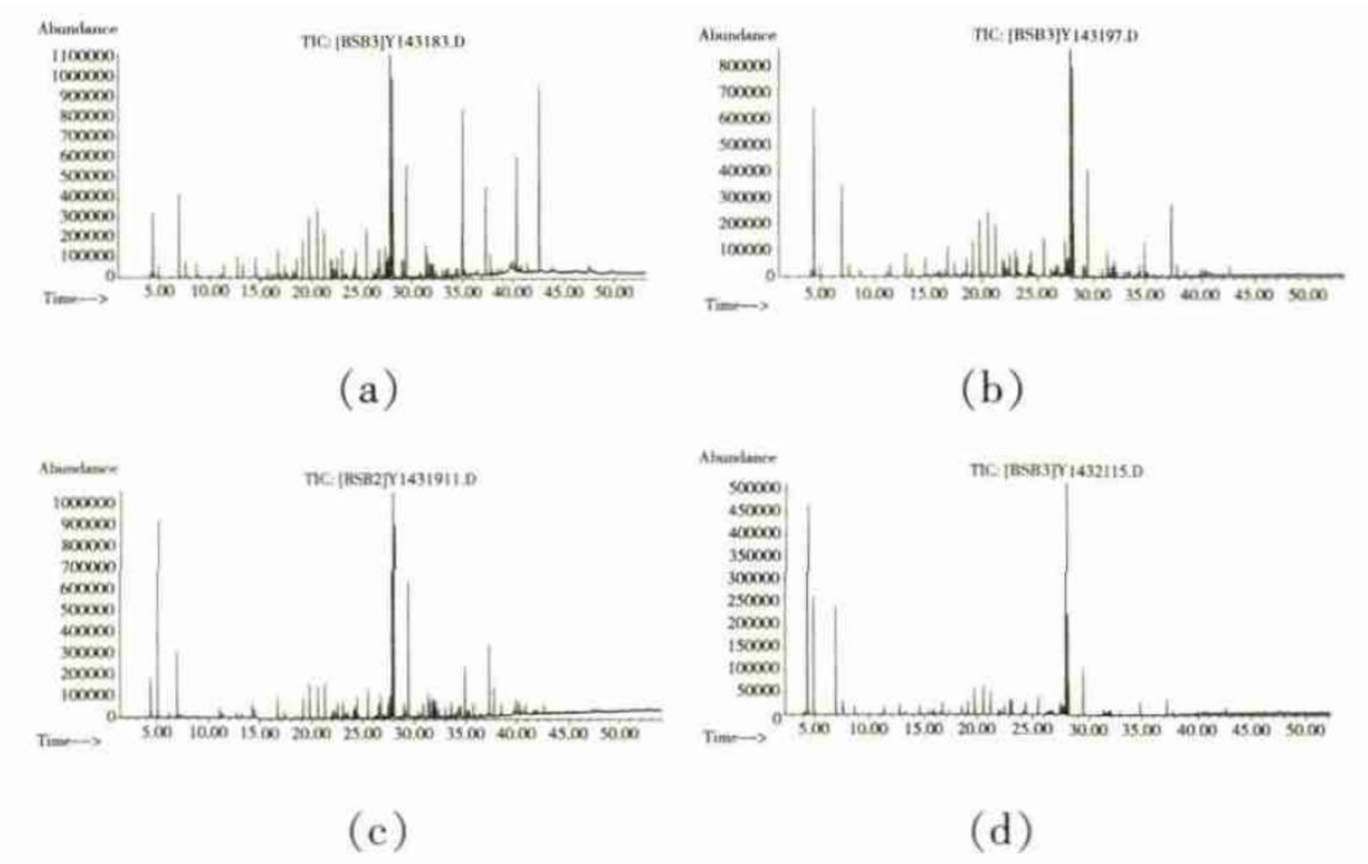

Figure 5. Total ion current diagram of corn stalk bio-oil at different particle sizes. **(a)** particle size 20–40 mesh; **(b)** particle size 40–60 mesh; **(c)** particle size 60–80 mesh; **(d)** particle size 80–120 mesh.

3.2 Effect of particle size of raw materials on the composition of corn straw pyrolysis bio-oil

The results of rapid pyrolysis test showed that when the pyrolysis reaction temperature was 500 ℃, the yield of bio-oil was the highest[13]. **Figure 5** is the GC-MS analysis results of four different raw material sizes when the reaction temperature is 500 ℃. The content of the main components is shown in **Table 3**. It can be seen that the particle size of the raw material has a certain effect on the composition of corn straw pyrolysis bio-oil, and the main components and their contents of the bio-oil are different.

In corn straw pyrolysis bio-oil, the content of 3-hydroxyl-2-phenyl-2-acrylic acid is the highest, which tends to change with the particle size of raw material, as shown in **Figure 6(a)**. The content of 3-hydroxyl-2-phenyl-2-acrylic acid is significantly affected by the particle size of the raw material at the same reaction temperature, and it increases with the decrease of the particle size, and the content is the highest when the particle size is 80–120 mesh. At the same time, the content of levoglucosan, an important component of bio-oil, also follows a similar law, and the content of raw material is the highest when the particle size is 80–120 mesh.

The raw material size 20–40 mesh bio-oil also contains about 3% hydroxysuccinic acid, but this substance was not detected in the biological oil with raw material of other particle sizes, indicating that the effect of particle size on the formation of hydroxysuccinic acid cannot be ignored. Similarly, the contents of methyl formate, diethyl phthalate, methyl pyridine carboxylate and dibutyl phthalate in bio-oil were higher when the particle size was 20–40 mesh, but with the decrease of particle size, these esters were not detected. In addition, the contents of 2-methoxy-4-vinyl phenol and 2.6-dimethoxyphenol in biological oil were higher, and with the decrease of raw material particle size, the content of 2-methoxy-4-vinylphenol and

2-dimethoxyphenol increased at first and then decreased, and the content was the highest when the particle size of raw material was 60–80 mesh.

Table 3. The main components comparison of corn stalk bio-oil at different particle sizes

Compound name	Peak area/%			
	20–40 mesh	**40–60 mesh**	**60–80 mesh**	**80–120 mesh**
Hydroxyacetaldehyde	10.51	10.06	4.15	11.32
Methyl formate	8.072	-	-	-
Acetic acid	1.88	1.64	0.85	2.84
Hydroxyacetone	2.69	2.11	-	3.66
Glycol	1.09	1.54	1.22	-
Methyl acetate	1.47	1.64	0.91	1.98
Butyraldehyde	1.32	1.68	1.57	2.10
2-furfuryl alcohol	-	1.65	-	1.44
2-hydroxy-2-cyclopentenone	1.06	-	1.64	1.00
2(5H)-furanone	0.98	1.11	0.98	1.15
Butyl TERT butylamine	-	1.47	1.51	-
3-methyl-2-hydroxy-2-cyclopentenone	2.43	2.76	2.82	2.51
Phenol	1.76	1.86	1.39	1.45
2-methoxyphenol	1.35	1.60	1.57	1.26
Glycerol	1.28	1.23	-	-
5-Butyl nonane	1.31	1.53	-	-
4-methylphenol	-	0.57	1.13	0.63
Cyclopentanol	1.55	1.26	1.06	1.87
Cyclopropyl methanol	-	0.90	1.92	0.70
4-ethyl phenol	1.11	0.99	1.02	0.83
3-methyl-2.4-furandione	1.24	1.24	3.54	1.93
Diethyl succinate	1.03	1.34	-	0.99
3-hydroxy-2-phenyl-2-acrylic acid	15.58	17.55	18.74	19.14
2-methoxy-4-vinyl phenol	4.70	5.73	7.10	4.53
Propyl butyrate	-	2.32	-	2.35
Hydroxysuccinic acid	2.16	-	-	-
5-hydroxy-2-furaldehyde	-	0.78	0.95	-
2.6-dimethoxyphenol	2.76	3.28	5.41	2.64
Hydroquinone	-	-	0.88	0.44
Octadecenoic acid methyl ester	-	0.75	2.69	-
3-tert-butyl-4-hydroxyanisole	-	1.27	2.40	-
Diethyl phthalate	4.62	-	-	-
Levoglucosan	1.54	2.71	4.01	4.57
Methyl picolinate	2.80	-	-	-
Dibutyl phthalate	7.95	-	-	-

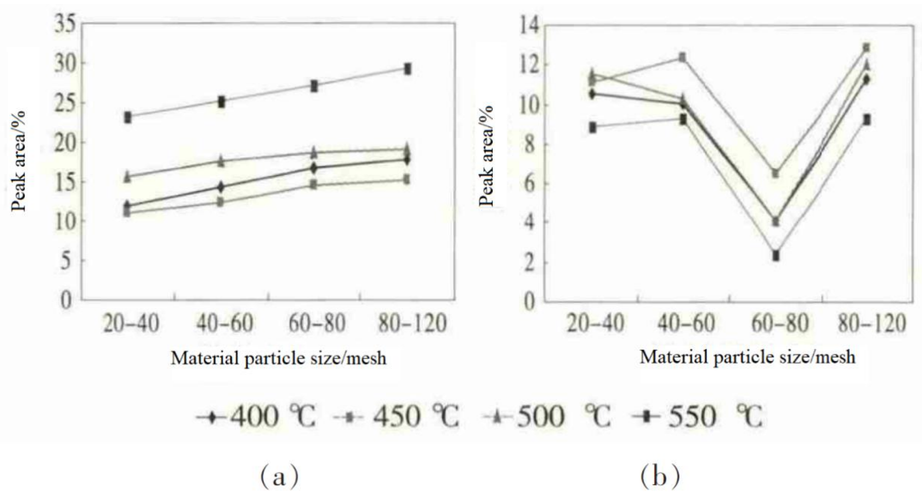

Figure 6. Relationship of bio-oil composition and particle size.
(a) 3-hydroxy-2-phenyl-2-acrylic acid; (b) Hydroxyacetaldehyde.

It can be seen from **Table 2** that hydroxyacetaldehyde is the highest content of aldehydes in the biological oil at 500 ℃. **Figure 6(b)** is the change of hydroxyacetaldehyde content in bio-oil obtained by pyrolysis under different conditions. It can be seen that the effect of raw material particle size on hydroxyacetaldehyde content is similar, its content is close to 20–40 mesh and 40–60 mesh, but it decreases obviously at 60–80 mesh, and slightly higher at 80–120 mesh than at 40–60 mesh. It can be seen that the particle size of raw materials has a significant effect on the formation of hydroxyacetaldehyde, which is not conducive to the formation of hydroxyacetaldehyde in bio-oil when the particle size is 60–80 mesh.

Figure 7 shows the content of each component in the biological oil obtained by pyrolysis of four kinds of corn straw powder at different reaction temperatures. it can be seen that the contents of acids and phenols are the highest, followed by esters and aldehydes, ethers and alkane compounds.

At the same reaction temperature, compared with the particle size of other raw materials, the content of ester compounds in bio-oil is 20% higher than that of other raw materials, and the content of ester compounds is very small and has no obvious change at the other particle sizes. It can be inferred that more ester compounds can be formed when the particle size of corn straw powder is 20–40 mesh.

By comparing the contents of bio-oil components at four reaction temperatures in **Figure 7**, it is found that the bio-oil also contains a large number of phenolic and aldehyde compounds, which are generally lower than those of acid compounds. At the same reaction temperature, when the particle size of raw material is 60–80 mesh, the content of phenolic compounds in bio-oil is the highest, while the content of aldehyde compounds is the lowest. It can be inferred that compared with other particle size ranges, when the particle size of corn straw powder is 60–80 mesh, it is more beneficial to the formation of phenolic compounds, but not conducive to the formation of aldehyde compounds.

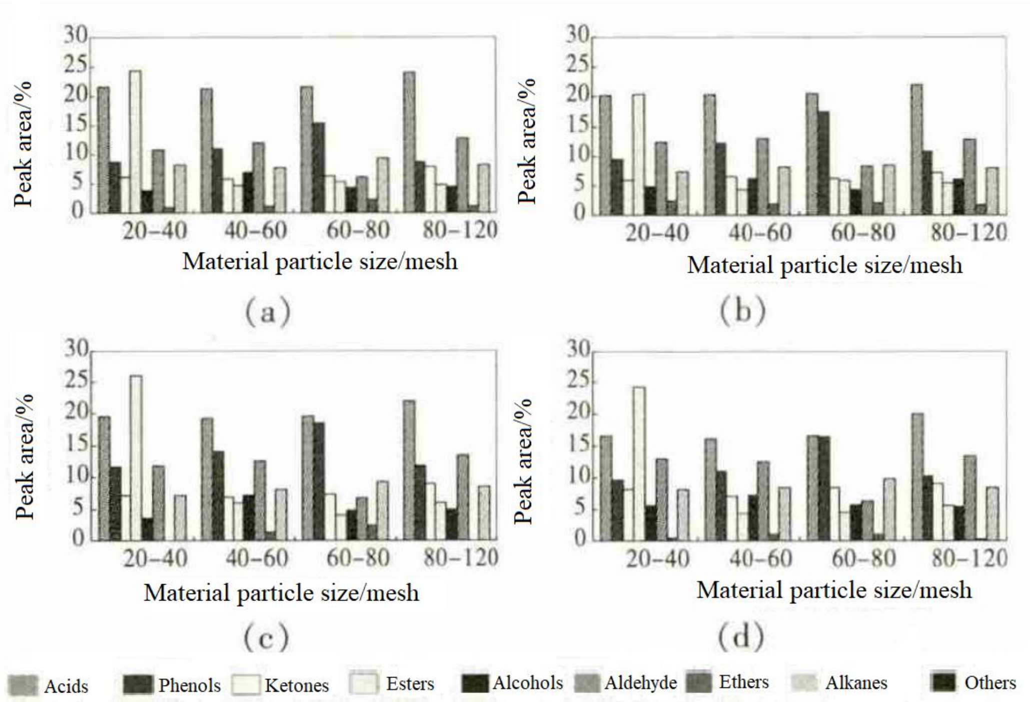

Figure 7. Distribution of the main chemical families of bio-oil. **(a)** reaction temperature 400 °C; **(b)** reaction temperature 450 °C; **(c)** reaction temperature 500 °C; **(d)** reaction temperature 550 °C.

In addition, the rapid pyrolysis bio-oil of corn straw powder also contains a small amount of ketones, alcohols, alkane and other kinds of compounds, the content of which is relatively small, and at the same reaction temperature, its content does not change obviously with the change of raw material particle size, which indicates that for corn straw powder, the particle size of raw materials has no significant effect on the formation of ketones, alcohols and alkane compounds in rapid pyrolysis bio-oils.

4. Conclusion

(1) Corn straw pyrolysis bio-oil contains a large amount of 3-hydroxyl-2-phenyl-2-acrylic acid, and it takes up small content of bio-oil when the reaction temperature is 450 ℃, but with the increase of reaction temperature, the total amount of acid compounds in bio-oil decreases; as the particle size of raw material decreases, the content of 3-hydroxyl-2-phenyl-2-acrylic acid increases.

(2) with the increase of reaction temperature, the content of hydroxyacetaldehyde in bio-oil increased at first increased and then decreased: when the particle size is 20–40 mesh, the content of hydroxyacetaldehyde is the highest at 500 ℃, and it is the highest at 450 ℃ when the particle size is the other three kinds; the content of hydroxyacetaldehyde and other aldehyde compounds was the lowest when the particle size of raw material was 60–80 mesh.

(3) The reaction temperature of 500 ℃ and the particle size of 60–80 mesh raw materials are beneficial to the formation of phenols in bio-oil, in which the contents of 2-methoxy-4-vinyl phenol and 2, 6-dimethoxyphenol are relatively higher.

(4) Compared with other particle sizes of raw materials, the content of ester compounds in bio-oil with particle size of 20–40 mesh is 20% higher than that of other raw materials, while the content of ester compounds in other particle sizes is lower and has little change.

(5) The reaction temperature and the particle size of raw materials had no significant effect on the formation of ketones, alcohols and alkane compounds in bio-oils.

Conflict of interest

The authors declared no conflict of interest.

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