Classification and comparison of physical and chemical properties of corn stalk from three regions in China

Wang Lu[1,2], Liu Ronghou[1,2*], Sun Chen[1], Cai Wenfei[1], Tao Yiwei[1], Yin Renzhan[1], Mei Yuanfei[1]

(1. *Biomass Energy Engineering Research Center, School of Agriculture and Biology, Shanghai Jiao Tong University, Shanghai 200240, China*; 2. *Key Laboratory of Urban Agriculture (South), Ministry of Agriculture, Shanghai 200240, China*)

Abstract: Corn stalk samples from Anhui, Jiangxi and Shanghai were used as test materials. Their physical, chemical and thermo-chemical engineering characteristics were analyzed. The similarities and differences in properties of corn stalk from the three regions were determined using SIMCA-P and SPSS software in order to obtain a proper energy utilization method of corn stalk. The results show that the corn stalk from Shanghai has significant differences from the samples of Jiangxi and Anhui. In particular, the following properties of corn stalk from Shanghai such as the contents of cellulose, calcium (Ca), iron (Fe), crude ash, volatile matter, carbon (C), nitrogen (N), and oxygen (O) are significantly different from those of Jiangxi and Anhui samples (P<0.05). While other properties such as the contents of magnesium (Mg), copper (Cu), zinc (Zn), moisture, hydrogen (H), and sulfur (S) have no significant difference among samples of three regions. Compared with the corn stalk in Anhui and Jiangxi, the Shanghai samples are more suitable for the production of ethanol because of their higher ratio of cellulose to hemi-cellulose content. Because of its high content of ash and low calorific value, the Shanghai corn stalk is suitable for the gasification process instead of for direct combustion or bio-oil production. The research can provide a reference for raw material selection for biomass energy production and utilization.

Keywords: corn stalk, physical and chemical properties, bioenergy, principal components analysis, partial least squares discriminant analysis

DOI: 10.3965/j.ijabe.20140706.012

Citation: Wang L, Liu R H, Sun C, Cai W F, Tao Y W, Yin R Z, et al. Classification and comparison of physical and chemical properties of corn stalk from three regions in China. Int J Agric & Biol Eng, 2014; 7(6): 98－106.

1 Introduction

Corn is an important source of feed and food with the highest yield among crops in the world. Moreover, corn stalk as an important biomass has also received a great attention in China and the rest of the world in recent years. Corn stalk has a variety of uses, such as fuel, fertilizer, feed, raw materials, etc.[1] Specifically, it is an important raw material for biomass energy production through several biomass energy conversion technologies such as thermo-chemical conversion technology including pyrolysis and gasification, and biological conversion technology including ethanol and methane fermentation[2,3]. Chinese organic fertilizer nutrient data shows that stalk of the same variety of corn in different regions has different nutrient contents and is used in different ways. As it is known, stalk composition will affect the efficiency and quality of biomass energy directly or indirectly[4]. For example, the main product of biomass fast pyrolysis is bio-oil. From the elemental analysis and the water content of the bio-oil, its molecular

Received date: 2013-10-08 **Accepted date:** 2014-11-07

Biographies: Wang Lu, MS, research focuses on biomass characteristic analysis. Email: 1990xianrenzhang@163.com. **Sun Chen,** PhD, research focuses on biomass characteristic analysis and biogas. Email: sunbeammy@163.com. **Cai Wenfei,** PhD, research focuses on production of bio-oil. **Tao Yiwei,** experimental engineer, research focuses on analytical chemistry. Email: taoyw@sjtu.edu.cn. **Yin Renzhan,** PhD, research focuses on pyrolysis and stability of bio-oil. Email: yinrenzhan@163.com. **Mei Yuanfei,** PhD, research focuses on the numerical simulation of pyrolyis and production of bio-oil. Email: meiyuanfei@foxmail.com.

***Corresponding author: Liu Ronghou,** PhD, Professor, Director, research interests: Biomass Energy Engineering. Mailing address: Biomass Energy Engineering Research Center, School of Agriculture and Biology, Shanghai Jiao Tong University, Shanghai 200240, China. Tel: +86 21-34205744. Email: liurhou@sjtu.edu.cn.

formula can be calculated[5]. Therefore, it is of significance to carry out the comparative study on the basic characteristics of corn stalk from different regions in order to provide reference for optimum energy-oriented utilization.

Many studies on the characteristics of corn stalk have shown that corn stalk as a biomass raw material can be used for the preparation of bio-char and bio-oil[6-8]. Through deeper research on the preparation technology and properties of the bio-char obtained from four kinds of biomass including pine cone, soybean cake, corn stalk and peanut shells, Apaydin-Varol and Pütün[9] found a positive correlation between chemical properties of bio-char and the content of three components of the stalk fiber. Moreover, notable differences in chemical properties of bio-char from different stalks also existed. Hou[10] studied the mechanical characteristics and physicochemical property of corn straw. The research on physical properties such as bulk density and frictional characteristics of corn stalk from different areas found that the difference in raw material accumulation feature of stalk from different regions or kinds should be fully considered in the design of related equipment and process routes, such as compression, transport and storage of stalk material[11]. Through the industrial analysis and the comparison of physical and chemical properties, moisture content, and calorific value of corn stalk from North and Northeast China, Tian et al.[12] found that physical and chemical properties of the corn stalk in different area were different. However, it lacks detailed comparison and analysis on the composition of corn stalk from different regions of South China. Therefore, the objectives of this research were to study the physical, chemical, and thermo-chemical characteristics of corn stalk from three regions including Bozhou city in Anhui, Jiujiang in Jiangxi, and Chongming Island of Shanghai, and to investigate the intrinsic correlation of corn stalk from the three regions in order to provide the basis for the selection of raw materials and choice of biomass energy conversion technology.

2 Materials and methods

2.1 Materials

Corn stalks including stem and leaves were collected from 21 different plots in Bozhou (Anhui, seven plots), Jiujiang (Jiangxi, five plots), and Chongming (Shanghai, nine plots). Two kilograms corn stalks were collected from each plot. According to geographical origins, corn stalks were divided into three groups and marked Anhui stalk, Jiangxi stalk and Shanghai stalk. After drying, corn stalks were smashed to branches and ground to powder. The powder samples were dried at 45℃, screened by 20 mesh sieves, and used for testing.

2.2 Composition measurement methods

Corn stalk powder sample 50 g was used for testing for the content of cellulose, hemicelluloses, lignin, moisture, crude ash, volatile matter, fixed carbon, C, H, N, S, O and some metal elements. Indicators are measured according to the Chinese National Standards, American Society for Testing and Materials Standards (ASTM) and Association of Official Analytical Chemists Standards (AOAC). Determination methods of various indicators are shown in Table 1.

Table 1 Determination methods of various indicators

Parameter	Standards
Cellulose Hemicelluloses	NREL/TP-510-42618 Determination of Structural Carbohydrates and Lignin in Biomass[13]
Lignin	ASTM E1721-01 Standard Test Method for Determination of Acid-insoluble Residue in Biomass[14]
P, K, Ca, Na, Mg, Fe, Cu, Zn	Metals in Plants and Pet Foods[15]
Moisture	ASTM E1756-08 Standard Test Method for Determination of Total Solids in Biomass[16]
Ash	ASTM E1755-01(2007) Standard Test Method for Ash in Biomass[17]
Volatile matter	ASTM E872 -82(2006) Standard Test Method for Volatile Matter in the Analysis of Particulate Wood Fuels[18]
Fixed carbon	ASTM E 870-82 Standard Test Method for Analysis of Wood Fuels [18]
C, H	ASTM E777 -08 Standard Test Method for Carbon and Hydrogen in the Analysis Sample of Refuse-Derived Fuel[19]
N	AOAC Official Method 990.03 Protein (crude) in Animal Feed[20]
S	ISO351:1996, Solid Mineral Fuels-Determination of Total Sulfur-High Temperature Combustion Method

2.3 Experimental apparatus

The following experimental apparatuses were used: Branches grinder (112M-4, made by Fulunlite in China), Chinese medicine grinder (DFY-500, made by Dade in China), analytical balance (ML104, made by Mettler in Switzerland), GPC (Waters 1515-2414, made by Waters in Massachusetts, U.S.A) and UV spectrophotometer (UV-1800, made by Shimadzu in Japan) to test cellulose, hemicelluloses and lignin, inductively coupled plasma–optical emission spectroscopy (ICP–OES) to test mineral metals (model: MIC IC, made by Metrohm in Switzerland), muffle furnace (SX2-2.5-10, made by Jiangdong in China) to test crude ash content, lignin and volatile matter content, Elementar (Vario EL Ⅲ / Isoprime, made in Germany) to test C, H, N, S.

2.4 Data analysis methods

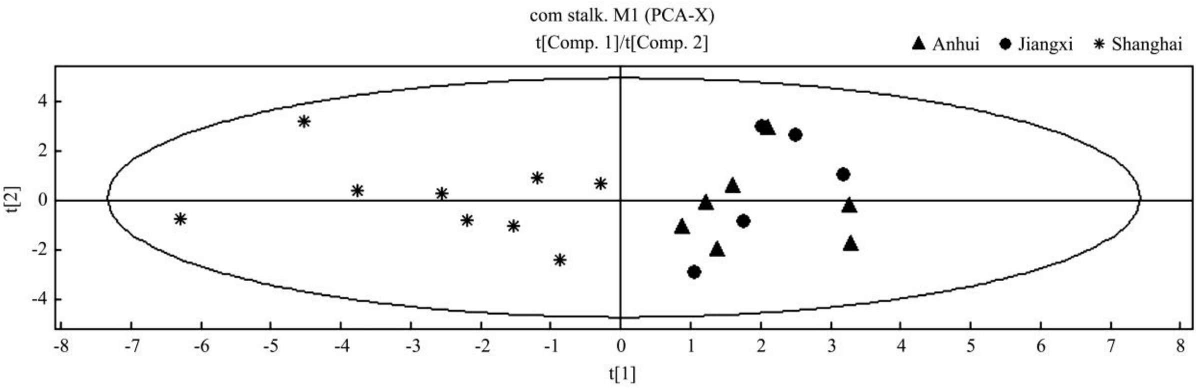
The data were exported to an Excel (Microsoft Excel 2007) table, and analyzed by principal components analysis (PCA) and partial least squares discriminate analysis (PLS-DA) using SIMCA-P software. PCA is used to analyze whether there are specific points. Then PLS can be used to view the differences between the two known substances. And variance analysis was carried out using SPSS software.

3 Results and discussion

3.1 Principal component analysis of corn stalk from three regions

Principal component analysis (PCA) is a mathematical procedure that uses orthogonal transformation to convert a set of observations of possibly correlated variables into a set of values of linearly uncorrelated variables called principal components. PCA in many ways forms the basis for multivariate data analysis[21]. Many parameters of corn stalk were tested. In order to investigate whether the characteristics of the corn stalk from three regions have difference or not, PCA is used. All test data of characteristics of 21 corn stalk samples were input to the PCA model. The two-component model was adopted.

Figure 1 shows the PCA score plot from the analysis of corn stalk. In Figure 1, t[1] and t[2] represent the vector of two main components, respectively. Its main purpose is to identify the specific points. Triangles represent corn stalk from Anhui, points represent that from Jiangxi and stars represent that from Shanghai. From Figure 1, it can be seen that the corn stalk from Shanghai has significant differences from Anhui and Jiangxi regions. However, no obvious difference of corn stalk was found between Anhui and Jiangxi.



Note: $R^2X[1] = 0.367865$, $R^2X[2] = 0.146789$, Ellipse: Hotelling T^2 (0.95). R^2X represents the explanation rate of the X variables on the X matrix variance; Ellipse: Hotelling T^2 (0.95) mean that the ellipse represents data at confidence interval of 95% by Hotelling T^2 test;t[Comp. 1] means t[1] and t[Comp. 2] means t[2], which represent the vector of two main components, respectively. t[Comp. 1]/t[Comp.2] mean the ratio of t[Comp. 1] to t[Comp. 2].

Figure 1 PCA scores plot from the analysis of corn stalk

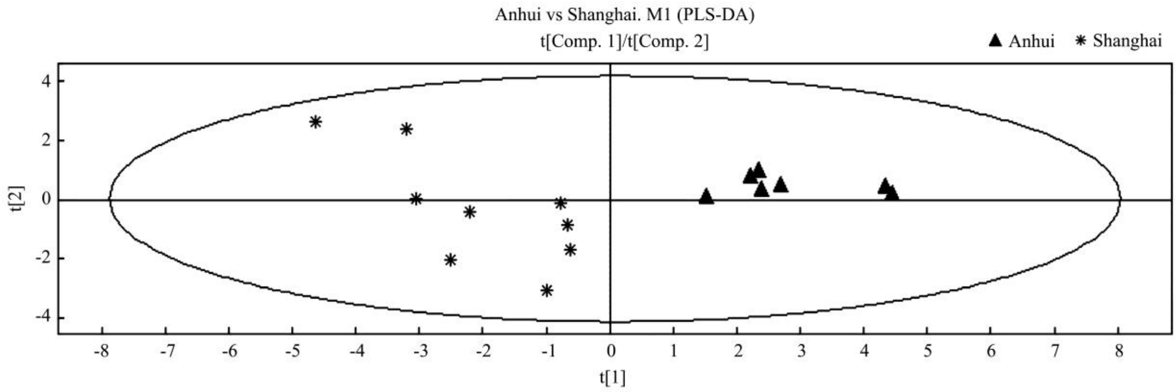
3.2 Partial least squares discriminant analysis (PLS-DA) on corn stalk from three regions

3.2.1 PLS-DA on corn stalk from Anhui and Shanghai

Partial least squares discriminant analysis (PLS-DA) is a PLS regression method with a special binary ‘dummy’ y-variable and is commonly used for classification purposes, analyzing mixture data and selecting biomarker in metabolomics studies[22].

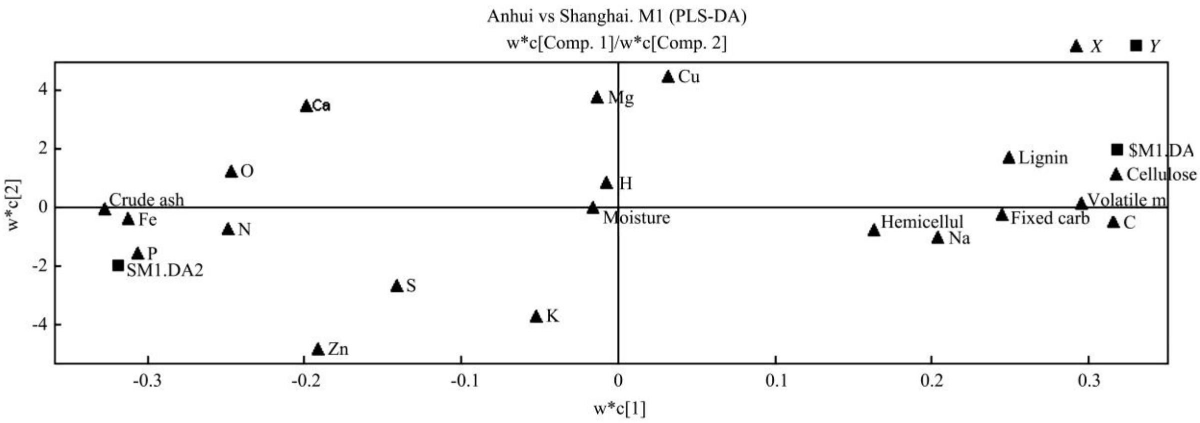
Analyzing mixture data with multiple regressions necessitates special model forms due to the mixture constraint. PLS-DA which works well in practice was used[23]. Figure 2 shows the PLS-DA score plot from the analysis of corn stalk from Anhui and Shanghai. The parameters ($R^2y = 0.963$, $Q^2 = 0.809$) obtained from the software indicated that the model presented was able to distinguish the properties between corn stalks from Anhui and Shanghai. Figure 3 shows loading plot of the PLS-DA model derived from corn stalks from Anhui and

Shanghai. In Figure 3, each point represents a kind of property. The farther distance between the point and the center point, the more variables contribute to the model. Therefore, from Figure 3, it can be inferred that parameters including the content of element C, P, Fe, cellulose, crude ash and volatile matters are the main factors that make a difference of corn stalk from Anhui and Shanghai.



Note: $R^2X[1] = 0.39978$, $R^2X[2] = 0.155464$, Ellipse: Hotelling T^2 (0.95).

Figure 2   PLS-DA score plot from the analysis on corn stalks of Anhui and Shanghai

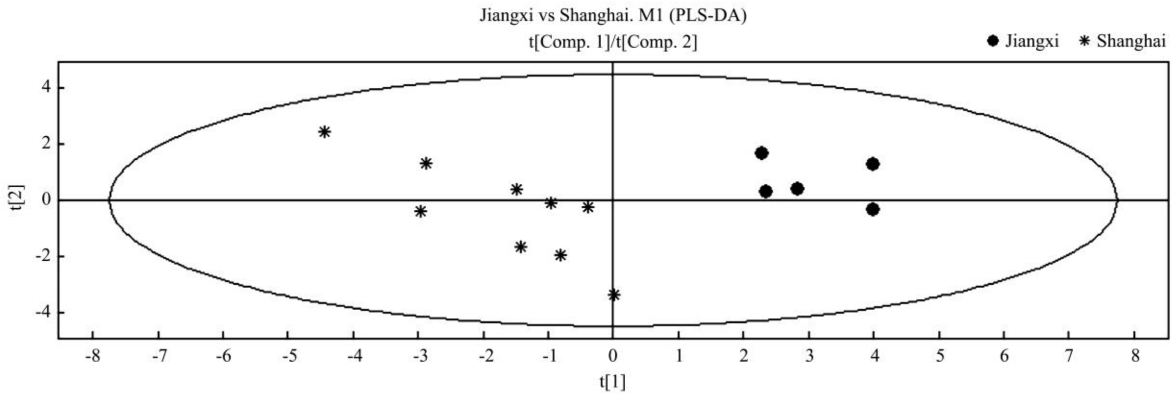


Note: $R^2X[1] = 0.39978$, $R^2X[2] = 0.155464$.   *X* represents normal values; *Y* represents abnormal values.

Figure 3   Loading plot of PLS-DA model derived from corn stalks of Anhui and Shanghai

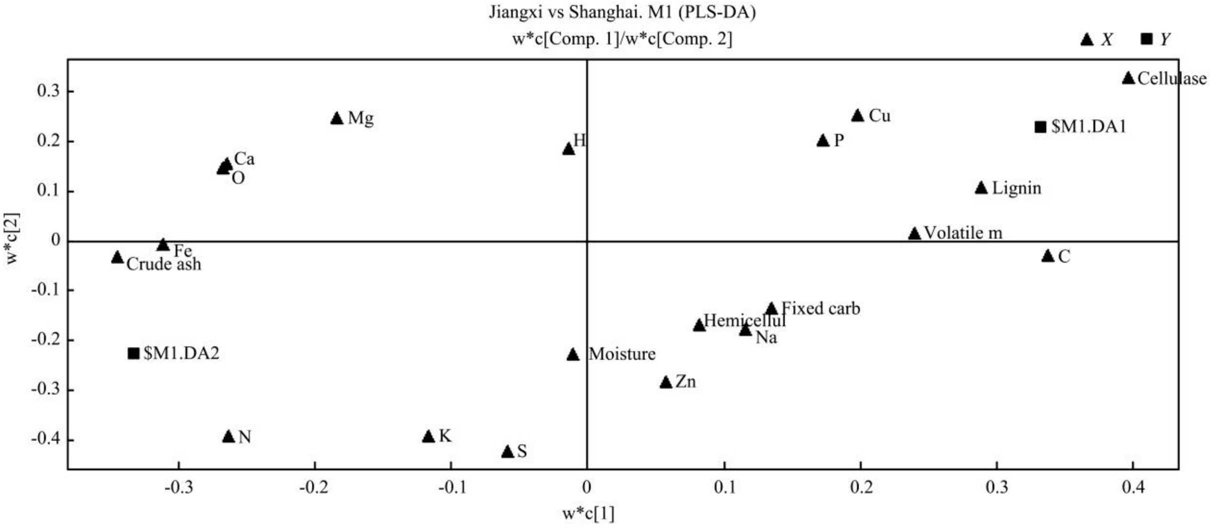
3.2.2   PLS-DA of corn stalk from Jiangxi and Shanghai

Figure 4 shows the PLS-DA score plot from the analysis of corn stalks from Jiangxi and Shanghai. Results of parameter ($R^2y = 0.982$, $Q^2 = 0.776$) obtained from the software indicated that the model presented was able to distinguish the properties between corn stalks of Jiangxi and Shanghai. Figure 5 shows the loading plot of PLS-DA model derived from corn stalks from Jiangxi and Shanghai. From Figure 5, it can be deduced that parameters including the content of element C, Fe, cellulose and crude ash are the main factors that distinguish corn stalks from Jiangxi and Shanghai.



Note: $R^2X[1] = 0.377769$, $R^2X[2] = 0.147901$, Ellipse: Hotelling T^2 (0.95).

Figure 4   PLS-DA scores plot from the analysis on corn stalks of Jiangxi and Shanghai



Note: $R^2X[1] = 0.377769$, $R^2X[2] = 0.147901$. *X* represents normal values; *Y* represents abnormal values.

Figure 5 Loading plot of the PLS-DA model derived from corn stalks of Jiangxi and Shanghai

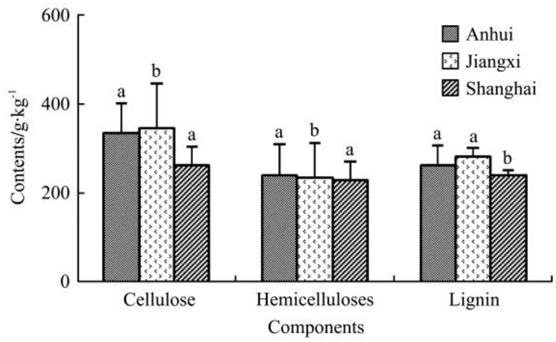
3.3 Properties of corn stalk

Furthermore, variance analysis was used through SPSS to validate the results of PCA and PLS-CA. After some outliers were removed, a variety of characteristics of corn stalk were classified and analyzed.

3.3.1 Fiber material

Fiber material includes cellulose, hemicelluloses and lignin. The level of fiber material content can be used as an important basis for biomass feedstock selection. The fiber content of stalks in three regions is shown in Figure 6. The average contents of the fiber of corn stalks from Anhui, Jiangxi and Shanghai are as follows: cellulose contents were 334, 345, 261 g/kg, respectively; hemicellulose contents were 239, 233, 226 g/kg, respectively; lignin contents were 260, 280, 237 g/kg, respectively. The contents of cellulose and hemicellulose determined in this research were similar to the report by Pasangulapati, et al.[25] in which the contents of cellulose and hemicellulose were 330-350 g/kg and 210-240 g/kg, respectively. The lignin content of our research is slightly higher than the above report in which the lignin content was 170-220 g/kg[25]. The contents of cellulose and lignin of the Shanghai stalk are both the lowest of the three.

The main products in the thermo-chemical reaction of cellulose and hemicelluloses are volatiles, while it is carbon for lignin[24]. According to Pasangulapati et al.[25], cellulose and hemicelluloses can be converted to CO, CO_2 in the thermo-chemical gasification reaction[26]. The conversion rate of carbon is higher for cellulose and hemicelluloses, while it is lower for lignin, which is mostly converted to CH_4. In practical applications, the choice of biomass conversion technology can be determined according to the content of cellulose, hemicelluloses and lignin. Figure 6 shows that the Jiangxi stalk is more suitable for the production of CH_4 among three regions because of its higher ratio of cellulose to hemi-cellulose.



Note: The same superscript letters indicate no significant difference in the same indicator (LSD P>0.05).

Figure 6 Fiber contents of corn stalks in three regions

For the production of ethanol, a biomass feedstock with a high average ratio of cellulose to hemi-cellulose content is needed. While the lignin content represents a potentially large energy source to increase the production of ethanol[27]. It can be calculated that the Shanghai stalk has the highest average ratio of cellulose to hemi-cellulose content at 0.87. The main raw materials for the production of ethanol are cellulose and hemicellulose. It is difficult to break down lignin.

Then it could be inferred that the Shanghai corn stalk is more suitable for producing ethanol.

3.2.2 Mineral metals

Mineral metals in stalk are mainly K, Na, Ca, Mg, Fe, Cu, Zn, etc. Contents of metal elements of corn stalks in three regions are shown in Table 2. Minerals are trace elements and essential for plant body. When the plants cannot absorb these elements sufficiently, they would be subjected to malnutrition; if the concentrations of minerals are too high, it would be easy to cause poisoning. K^+ is the most abundant cation for plants. According to Table 2, the content of metal elements such as Mg, Cu, and Zn has no significant difference in three regions. Anhui stalk has the highest average level of Na (2.028 g/kg). Shanghai stalk has the highest average levels of Ca (6.713 g/kg) and Fe (3.230 g/kg), which agrees with the result of PLS-DA. It is likely because the samples of Shanghai of our research were taken from Chongming Island. Corn in Chongming, Shanghai grows from March to August when it is warm and moist in the island. Corn in Anhui and Jiangxi grows from June to October when it is arid in the inland. The climate conditions between island and inland have a great difference during corn growth period. In addition, the reason that the content of Fe of corn stalk in Chongming Island is higher is probably the higher content of efficient iron in soil (more than 20 mg/kg), which is much higher than those in Jiangxi and Anhui. Xu et al.[28] reported that the effective Fe content in the soil of Chongming Island of Shanghai is above average.

Table 2 Contents of metal elements of corn stalks in three regions

		$K/g\cdot kg^{-1}$	$Na/g\cdot kg^{-1}$	$Ca/g\cdot kg^{-1}$	$Mg/g\cdot kg^{-1}$	$Fe/g\cdot kg^{-1}$	$Cu/mg\cdot kg^{-1}$	$Zn/mg\cdot kg^{-1}$
Anhui	mean	15.283^a	2.028^a	4.566^a	4.108^a	0.407^a	0.016^a	0.029^a
	SD	6.133	0.652	1.509	0.677	0.166	0.005	0.012
	MAX	26.420	2.715	7.056	5.300	0.654	0.023	0.048
	MIN	7.129	1.095	2.728	3.415	0.205	0.009	0.014
Jiangxi	mean	12.615^a	1.706^{ab}	3.686^a	2.745^a	0.506^a	0.022^a	0.124^a
	SD	6.144	0.599	0.968	0.874	0.234	0.009	0.125
	MAX	23.430	2.478	4.639	3.697	0.751	0.031	0.344
	MIN	8.413	0.830	2.598	1.760	0.160	0.012	0.046
Shanghai	mean	17.333^a	1.450^b	6.713^b	4.197^a	3.230^b	0.015^a	0.097^a
	SD	9.470	0.383	2.276	1.792	1.572	0.006	0.087
	MAX	28.870	2.044	10.410	8.369	5.644	0.029	0.247
	MIN	5.401	0.960	4.624	2.527	1.027	0.007	0.031

Note: The same superscript letters indicate no significant difference in the same column (LSD, $P>0.05$).

Mineral metals have great impact on biomass energy utilization. McKendry[27] pointed out that the alkali metal element has played a more important role in thermo-chemical conversion process. Wang et al.[29] reported that acid wash can decrease the concentration of alkaline cations in biomass, enhance the mass loss rate of biomass pyrolysis, and shift the pyrolysis to higher temperature. Alkaline compounds addition can promote biomass pyrolysis at lower temperature.

3.3.3 Proximate analysis

Proximate analysis generally includes moisture, crude ash, volatile matter and fixed carbon. The samples were dried at 45℃ before test. Proximate analysis of corn stalk in three regions is shown in Figure 7. Volatile matter is an important indicator for fuel classification. Fixed carbon is the yield of an inorganic substance left over after determination of volatile. Fixed carbon can be calculated according to the following formula:

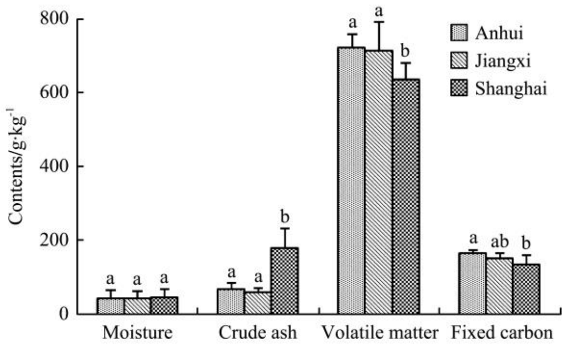
$$Fc = 1000 - Wc - CAc - Vc \tag{1}$$

where, Fc is fixed carbon, g/kg; Wc is water content, g/kg; CAc is crude ash content, g/kg; Vc is volatile matter content, g/kg.

Results of volatile matter contents of corn stalks from Anhui, Jiangxi, and Shanghai were 650-757, 583-776, 579-713 g/kg, respectively. Tian et al[12] reported that average of volatile matter content is 700 g/kg in north of China. The result in this paper is close to Tian's report.

Figure 7 shows that Anhui, Jiangxi and Shanghai stalks have little difference in moisture content. But for Shanghai stalk, the content of crude ash was significantly higher than that of stalks from other regions, while volatile matter was significantly lower than that of stalks from other regions. It shows that the trend of the fixed carbon content of stalks from three regions is as follows:

Anhui stalk > Jiangxi stalk > Shanghai stalk. And the higher ash content in biomass was disadvantageous to obtain high quality bio-oil product[30]. Therefore, compared to Anhui and Jiangxi stalks, Shanghai corn stalk is not suitable as raw material for the production of bio-oil by pyrolysis, and bio-oil would age easier[31].



Note: The same superscript letters indicate no significant difference in the same indicator (LSD P>0.05).

Figure 7 Proximate analysis of corn stalks in three regions

3.3.4 Inorganic elements

Inorganic elements in biomass include C, H, N, S, O and P, etc. Contents of inorganic elements in three regions of corn stalk are shown in Table 3. The results showed that contents of H and S among Anhui, Jiangxi and Shanghai stalk have little differences. Shanghai stalk is significantly different from stalk of other regions in C, O and N contents. This is because during the growth period of corn, the atmospheric temperature is high and it is rainy in Chongming Island. To a certain extent, it would inhibit photosynthesis of corn, resulting in the lower C storage capacity. The average P content is Jiangxi > Shanghai > Anhui.

C, H and S are combustible substances of biomass. Lower calorific value of biomass can be estimated according to the content of them in the biomass[32]. The formula[33] is as follows:

$$Q = 4.19[81C + 246H - 26(O - S)] - 6W \qquad (2)$$

where, Q is lower calorific value, kJ/kg; C, H, O, S, and W are fuel carbon, hydrogen, oxygen, sulfur and moisture content, respectively, %.

Biomass contains nitrogen and oxygen. Nitrogen cannot be burned to produce heat. Although oxygen does not emit heat, it can enhance the combustion reaction. These two elements will reduce heating value of biomass. Results of Table 3 show that Shanghai corn stalk has the minimum average lower calorific value of 13,976 J/g. And a higher N content of 16.08 g/kg of Shanghai corn stalk would result in more serious air pollution when it is burned. So Shanghai corn stalk is not fit for direct combustion utilization.

Table 3 Content of inorganic elements in three regions

		C/g·kg^{-1}	H/g·kg^{-1}	N/g·kg^{-1}	S/g·kg^{-1}	O/g·kg^{-1}	P/mg·kg^{-1}
Anhui	mean	435.59^a	58.63^a	11.36^a	3.46^a	463.51^a	1.02^a
	SD	8.63	6.03	3.20	0.61	9.29	0.46
	MAX	452.17	68.91	14.94	4.17	476.91	1.61
	MIN	425.55	52.32	6.73	2.40	448.67	0.43
Jiangxi	mean	441.59^a	58.50^a	11.84^a	3.72^a	459.86^a	3.08^b
	SD	8.67	4.71	1.75	0.23	5.05	0.89
	MAX	454.54	65.28	13.80	4.00	467.36	4.15
	MIN	434.69	52.82	10.26	3.49	453.38	2.14
Shanghai	mean	391.46^b	58.82^a	16.08^b	3.81^a	494.38^b	2.4^b
	SD	22.74	5.32	3.11	0.37	26.35	0.71
	MAX	423.48	67.42	20.95	4.37	526.53	3.81
	MIN	352.48	52.81	12.05	3.35	449.26	1.41

Note: The same superscript letters indicate no significant difference in the same column (LSD P>0.05).

4 Conclusions

1) The data analysis software SIMCA-P and SPSS gave almost identical results, which shows that the PCA and PLS-DA methods can be used for classification and comparison of biomass characteristics.

2) The corn stalk from Shanghai has significant differences from samples from the other two regions. Its C content is lower, and the Fe content higher, possibly because its origin in Chongming Island, a county of Shanghai where the climate conditions are vastly different from those of inland regions. The growth

period of corn in Chongming Island, Shanghai is from March to August when it is warm and moist. Corn in inland provinces Anhui and Jiangxi grows from June to October when it is arid.

3) Compared with the stalks in Anhui and Jiangxi, ash and oxygen contents of the Shanghai corn stalk are the highest and the C content is the lowest. So Shanghai corn stalk is not suitable for the production of bio-oil and direct combustion utilization, but suitable for gasification and production of ethanol because of the higher ratio of cellulose to hemi-cellulose content. Compared with the stalks in Anhui and Shanghai, the Jiangxi stalk is more suitable for the thermo-chemical gasification reaction.

Acknowledgements

We acknowledge that this work was financially support by Special Fund for Agro-scientific Research in Public Interests (Grant 201003063-09). We also would like to thank Instrumental Analysis Center of Shanghai Jiao Tong University for providing MIC IC, ICP-OES, Vario EL Ⅲ, Isoprime elemental analysis, and isotopic ratio mass spectrometry.

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