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Effects of Returning Granular Corn Straw on Soil Humus Composition and Humic Acid Structure Characteristics in Saline-Alkali Soil

Xiaodong Chen, Jinggui Wu * and Yaa Opoku-Kwanowaa

College of Resources and Environment Sciences, Jilin Agricultural University, Changchun 130118, China; chenxiaodongyes@126.com (X.C.); yokwanowaa@gmail.com (Y.O.-K.)

* Correspondence: wujinggui@jlau.edu.cn

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Abstract: Returning straw plays an important role in protecting the environment and maintaining the sustainable development of agriculture. In this research, we explored the effects of returning granular corn straw on soil humus composition and humic acid structural characteristics in a typical saline-alkali soil. This study was conducted in Jilin province. A randomized block design consisting of nine plots with three treatments in three replicates was used for this study. The treatments were granular corn straw (GS), coarse corn straw (CS), and control (CK). The results show that treatments GS and CS both increased the contents of soil humus and its components in the soil compared to CK. However, treatment GS recorded the highest significant increase in soil humus carbon (HEC), fulvic acid carbon (FAC), and humic acid carbon (HAC) by 17.59%, 8.32%, and 26.51%, respectively. Comparing the two straw treatments, it was found that the relative intensities of treatment GS at 2920 and 2850 cm^{-1} were higher than treatment CS by 1.58% and 72.49%, respectively. The relative intensities of treatment GS at 1720 cm^{-1} and 1620 cm^{-1} were lower than treatment CS by 52.2% and 30.43%, respectively. Moreover, an analysis of soil humic acid (HA) through an infrared spectrum, fluorescence spectrum, and principal components analysis (PCA) showed that the application of straw makes the structure of HA aliphatic, simple, and younger, and also promotes the continuous renewal of humus. In this study, the application of granular corn straw effectively improved the soil humus content and humic acid structural characteristics and is thus highly recommended.

Keywords: fluorescence spectrum; fourier transform infrared spectroscopy (FTIR) spectrum; humic acid; principal components analysis; granular corn straw; primary saline-alkali soil

1. Introduction

Corn straw is the residue left after harvesting corn. It contains a lot of organic matter and nutrients, which serve as a valuable biological resource [1]. The annual total amount of corn straw in China is huge, but the utilization rate is low [2]. In some instances, there is improper utilization, which causes a waste of resources and environmental pollution [3]. Therefore, it is better to return corn straw to the field to be applied as fertilizer since it can enhance soil fertility by increasing soil organic matter content (SOM), which is the major source of nutrients required for plant growth and the energy source for microbial activities [4]. SOM plays a significant role in soil fertility, environmental protection, and sustainable agricultural development [5]. Soil humus (HS) is an important part of SOM, which can be effectively increased with the application of straws [6]. Soil humus is mainly composed of soil humic acid (HA) and soil fulvic acid (FA). It is a component of soil organic matter that happens to be highly available in the soil, easily decomposed and mineralized by soil microbes, and directly affects nutrient supply in plants. It plays a key role in maintaining soil fertility, improving soil quality, and maintaining soil carbon pool balance [7].

HA forms the main constituent of humus (HS) and any changes in the structure and number of HA are closely associated with soil fertility [8]. Many studies have shown that there are many functional groups on the surface of HA molecules [9–11]. These functional groups have strong potentials to bind with soil nutrients, which can effectively retain soil nutrients and reduce nutrient loss in the soil [10]. HA also plays an important role in soil environmental health by influencing the transport and passivation of heavy metals in the soil [12]. Numerous studies have shown that returning crop straws, such as corn straws, to the field can affect the content and structure of humic acid in the soil [13,14]. In China, the main ways of returning straw include applying it as mulch, plowing the straw into the soil, and burying the straws deep into the soil [15]. However, these modes of returning straw are not effective in paddy fields since the straw can easily float when applied as mulch. Additionally, burying the straw deep in paddy fields is difficult to operate, involves high costs, and, most importantly, delays the decomposition process due to reduced soil aeration and microbial activity [16]. Therefore, in this experiment, the straw was returned to the field (0–20 cm depth) by shortening the length of the straw, which can accelerate the decomposition of straw, and also reduce carbon loss as the small straws can be protected by soil aggregates and clay particle [17].

Different types of soil respond differently to the application of organic materials [18]. The native saline-alkali soil is characterized by thin tillage, poor nutrients, and high salinity [19]; thus, applying corn straw for organic fertilization is very important in improving the fertility of native saline soil. However, few studies have been done to evaluate the effects of soil organic fertilizer on saline-alkali soil, especially to evaluate whether returning corn straw can improve the fertility of saline-alkali soil and its effectiveness in enhancing the humus content of the soil.

The purpose of this paper is to evaluate the changes in HA content and the structure of native saline-alkali soils under rice-planting conditions after corn straw application. The results of the study will help to provide knowledge on the effects of returning corn straw on soil humus and its related components. The results will, again, provide a basis for comparing the differences in the effects of different shapes of corn straw to ensure the sustainable use of straw in agricultural production and protecting the environment. We hypothesized that corn straw application increases the content of soil humus and its related components, while different shapes of straw can also have different effects on soil humus and its related components.

2. Materials and Methods

2.1. Experimental Site

This study was conducted in Haituo, Jilin province, which is located in Daan City (124°1′48″ E, 45°19′47″N). The study site is dry and has a temperate continental monsoon climate. The average annual precipitation, temperature, and the frost-free period are approximately 413.7 mm, 4.3 °C, and 157 d, respectively. The average yearly evaporation potential is 1702.44 mm, which is four times the average annual precipitation in the region. The average annual sun exposure is 3012.8 h, while the effective accumulated temperature of ≥10 °C is about 2921 °C [20]. The soil type is a typical halosol (classified according to the international soil taxonomy system). The drought and the saline-alkali nature of the soil creates an unsuitable condition for agricultural development. The main soil nutrients and properties of the study area are listed in Table 1.

Table 1. The main soil properties of the study area.

Soil Alkali Hydrolyzable Nitrogen ($g\cdot kg^{-1}$)	Organic Matter ($g\cdot kg^{-1}$)	Available Phosphorus ($mg\cdot kg^{-1}$)	Available Potassium ($mg\cdot kg^{-1}$)	Total Salt ($g\cdot kg^{-1}$)	pH	Clay %	Silt %	Sand %
11.28 ± 0.02	2.91 ± 0.03	20.61 ± 0.04	143.33 ± 2.32	3.39 ± 0.03	9.94 ± 0.05	40.5%	18.3%	41.2%

Note: Values are mean ± standard deviation.

2.2. Experimental Design

In this experiment, field studies were conducted in Haituo town for three years. A randomized block design consisting of nine plots with three treatments in three replicates was used for this study. The area of each plot was 6×5 m^2 and the treatments were granular corn straw (GS), coarse corn straw (CS), and control (CK). The granular corn straw was produced by breaking down the corn straw using a high-efficiency granulator and finally compacting them into 3–5 cm particles. The working motor power of the machine was 132 KV, the granulation temperature was 80 °C, and the granulation pressure was 606 kPa (Figure 1). Properties of the corn straws used are presented in Table 2. In total, the amount of corn straw that was applied to the field was adjusted in order to control the carbon to nitrogen ratio and also to prevent or solve the problem of nitrogen depletion. Hence the standard straw application rate (7500 kg/ha) was applied in this study. All organic materials (corn straws) were applied once in May 2016 before sowing the rice.

Figure 1. Picture showing the treatments used in this study; (**a**) coarse corn straw (CS), (**b**) granular corn straw (GS).

Table 2. Basic properties of organic materials.

Materials	Organic Matter (g·kg^{-1})	Total Nitrogen (g·kg^{-1})	Total Phosphorus (g·kg^{-1})	Total Potassium (g·kg^{-1})	pH
Corn Straw and Granular Straw	493 ± 2.16	8.33 ± 0.07	1.12 ± 0.02	12.3 ± 0.12	6.42 ± 0.08

In June 2018, soil samples were collected in 0–20 cm depth using a manual soil coring tube. Five sampling points were randomly selected in each plot, and soil samples were taken using the "S" method. Soil samples from each plot were combined and thoroughly mixed to form composite soils followed by air-drying. After air-drying the samples, stones and crop residues were removed, and the samples were sieved through a 2-mm mesh sieve.

2.3. Sample Determination

2.3.1. Extraction and Analysis of Humus

The composition of soil humus was analyzed following the method described by Dou [21]. The content of C in the humic substance was also determined using the $K_2Cr_2O_7$ external heating method [22]. The E4/E6 ratio of soil HA was calculated as the ratio of the absorbances of solutions at 465 nm and 665 nm [23]. The △logK value of HA was the absorbances of solutions in 400 nm and 600 nm, which was calculated as logK 400 nm/logK 600 nm.

2.3.2. Isolation and Purification of HA

The HA was isolated and purified following the method described by the International Society of Humic Substances [24]. The general steps involved are as follows: (1) decalcifying the soil with HCl; (2) leaching the decalcified soil with NaOH solution, followed by centrifuging. The resulting supernatant was HE. (3) The HE solution was then acidified to pH 1.0 and precipitated to HA after acidification. (4) The obtained HA was purified by dialysis. After lyophilization, the next relevant measurement was performed.

2.3.3. Infrared Spectrum Analysis

The HA infrared spectrum was analyzed by using the American AVATAR 360 Fourier transform infrared spectrometer. Using a microbalance, 0.5~2 mg of the HA sample was weighed, pulverized, and ground to <2 μm. The sample and KBr powder were mixed together in an agate mortar at a ratio of 1:100. The resulting mixture was pressed and measured in the wavelength range of 4000 to 500 cm^{-1}. The infrared spectrum was finally analyzed by Omnic software [25]. Following the research conducted by Senesi et al. [26] and Zhang et al. [27], the major series bands were assigned as follows: where 2920 cm^{-1} represents an asymmetric aliphatic CH stretching vibration peak, 2850 cm^{-1} represents a -CH_2- symmetric aliphatic CH stretching vibration peak. The band at approximately 1720 cm^{-1} was assigned to C=O stretching of COOH. The band, at approximately 1620 cm^{-1}, was assigned to aromatic C=C vibrations, H-bonded C=O of conjugated ketones, and C=O stretching of the amide groups.

2.3.4. Principal Component Analysis (PCA)

Usually, SPSS 16.0 software is used to reduce the dimensionality of the infrared spectrum data during PCA analysis. The PCA analysis helps to linearly transform multiple variables in order to select a small number and important variables for the establishment of a minimum data set. Multivariate statistics were used to multiply the indicators [28].

2.3.5. Fluorescence Emission Spectrum Analysis

The fluorescence emission spectrum of the soil humic acid sample was measured on a Hitachi F4500 fluorescence spectrophotometer with a wavelength range of 400–800 nm and a wavelength frequency of 5 nm. Finally, the software for the Hitachi F4500 fluorescence spectrophotometer was used to analyze the characteristic peaks of the spectral lines [29].

2.4. Data Analysis

The data collected were recorded and summarized in Excel 2003. Using SPSS version 16.0, the data collected was subjected to Variance Analysis (ANOVA) after performing a correlation analysis. The statistical significance level was used at 5% to test the variation between treatments. Origin software version 9.0 was used to present the results.

3. Results

3.1. Humus Content and Composition

Table 3 shows the organic carbon content of the soil humus components in each treatment. Comparing treatment CS with treatment CK, treatment CS recorded a significant increase in soil humus carbon (HEC), fulvic acid carbon (FAC), and humic acid carbon (HAC) by 34.68%, 19.43%, and 53.55%, respectively. Comparing treatment CK with treatment GS, treatment GS significantly increased the HEC, HAC and, FAC by 58.36%, 94.26%, and 29.36%, respectively. Comparing treatment GS and CS with CK, the HAC/FAC of treatment CS and treatment GS increased by 28.59% and 50.12%, respectively. This indicates that the content of soil humus increased with the application of corn straw. This helps to promote the degree of polymerization of the soil humus and also increases the degree of humification. Comparing the two corn straw treatments, the results show that treatment GS recorded a significant increase in HEC, HAC, FAC, and HAC/FAC by 17.59%, 26.51%, 8.32%, and 16.75%, respectively, as compared to treatment CS. As such, significant differences were observed in the effects of different shapes of the same organic materials on soil humus, where granular corn straw significantly increased the soil humus content.

Table 3. Soil humus composition relative to treatments.

Treatments	HEC ($g \cdot kg^{-1}$)	HAC ($g \cdot kg^{-1}$)	FAC ($g \cdot kg^{-1}$)	HAC/FAC
CK	0.819 ± 0.023 c	0.366 ± 0.011 c	0.453 ± 0.015 c	0.808 ± 0.021 c
CS	1.103 ± 0.027 b	0.562 ± 0.013 b	0.541 ± 0.014 b	1.039 ± 0.014 b
GS	1.297 ± 0.022 a	0.711 ± 0.018 a	0.586 ± 0.017 a	1.213 ± 0.022 a

Note: Values with different lowercase letters denote significant differences among different treatments at 5% levels.

3.2. E_4/E_6 Ratios and △logK Values of HA

The E4/E6 ratios of HA corresponding to treatment GS and CS were 15.22% and 3.8% higher ($p < 0.05$) than treatment CK, respectively, as shown in Figure 2. The △logK values of HA under soils treated with GS and CS were 32.43% and 13.51% higher, respectively, ($p < 0.05$) than the control (CK). Comparing the two straw treatments, the E4/E6 ratio under treatment GS was 10.99% higher ($p < 0.05$) than treatment CS. The △logK value of HA for treatment GS was 16.67% higher ($p < 0.05$) than treatment CS.

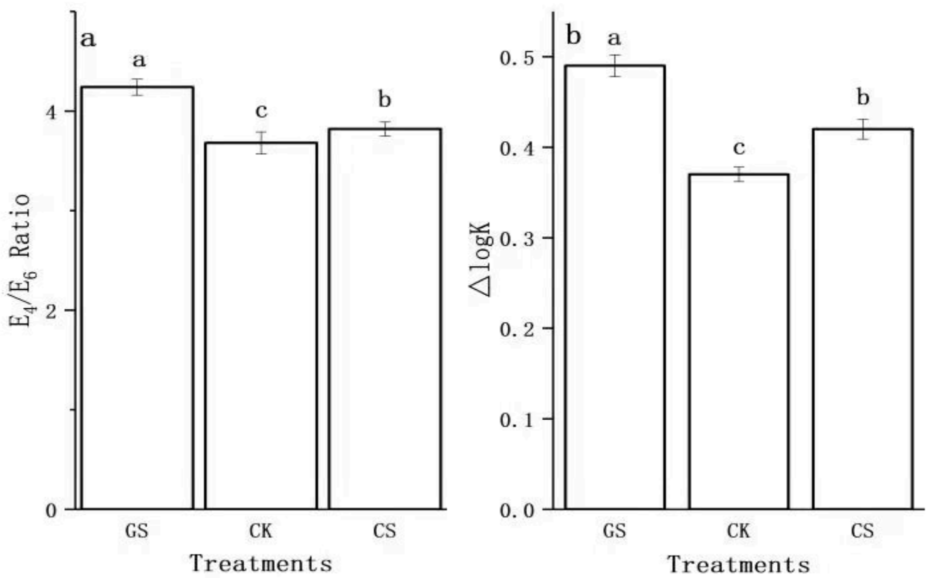

Figure 2. The E_4/E_6 ratios and △logK values of humic acid (HA) under the different treatments.

3.3. FTIR Spectrum of HA

Figure 3 shows the fourier transform infrared spectroscopy (FTIR) spectrum of soil HA. The infrared spectra of each treatment were similar in shape and structure, but the intensity of the absorption peaks of some key functional groups was different. The difference in the relative intensity of the absorption peaks of these infrared spectra also indicates that the application of different shapes of corn straws led to the difference in the presence of soil HA.

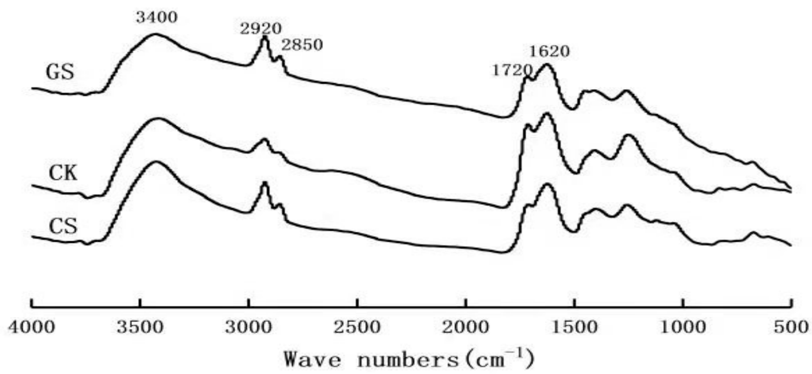

Figure 3. The FTIR spectrum of soil HA in the different treatments.

Comparing treatment GS and CK, GS recorded higher relative intensities of the aliphatic group bands at 2920 and 2850 cm^{-1} with increasing rates of 87.99% and 85.44%, respectively. This indicates that returning straw into the soil increases the relative content of aliphatic compounds in soil HA. Comparing treatment GS and CS with treatment CK, treatment GS and CS had a significant increase in the relative intensities at 1620 cm^{-1}, with an increasing rate of 14.51% and 64.61%, respectively. This indicates that returning the straw increased the content of aromatic carbon in soil HA. However, at 1720 cm^{-1}, the relative intensities of treatment GS and CS were lower than treatment CK, with a decreasing rate of 63.78% and 24.22%, respectively. This shows that the application of straw reduces the relative content of carboxyl groups in soil HA. Comparing the two straw treatments, it was found that the relative intensities of treatment GS at 2920 and 2850 cm^{-1} were significantly higher than treatment CS by 1.58% and 72.49%, respectively. The relative intensities of treatment GS at 1720 cm^{-1} and 1620 cm^{-1} were lower than treatment CS by 52.2% and 30.43%, respectively.

3.4. PCA of Soil HA Infrared Spectroscopy

The principal component analysis was performed on the infrared absorption spectra of the three treatments. The absorbance of the three treatment samples on all wavenumber segments was used as a variable, and all sample data were clustered by principal component analysis. The results are shown in Table 4. From Table 4, the results showed that principal component 1 accounted for 86.532% of the total variance contribution rate while principal component 2 accounted for 13.462%. The cumulative contribution rate of these two principal components accounts for 99.994% of the total variance contribution rate. This indicates that the use of these two main components can better represent the original spectrum. The main positive peaks of PC1 in Figure 4a are 2920 cm^{-1}, 1620 cm^{-1}, which are the absorption peaks of asymmetric aliphatic carbons and aromatic carbons respectively. The main positive peaks of PC2 in Figure 4b are 2920 cm^{-1}, 2850 cm^{-1}, and 1620 cm^{-1}, which represents asymmetric aliphatic carbons, symmetric aliphatic carbons, and aromatic carbon absorption peaks respectively. The results showed that the application of corn straw serves as one of the main sources of aliphatic carbon and aromatic carbon in the soil.

Table 4. Principal component, eigenvalues, and contribution rate results.

PC	Eigenvalues	Contribution Rate%	Accumulated Contribution Rate%
PC1	4.327	86.532	86.532
PC2	0.613	13.462	99.994

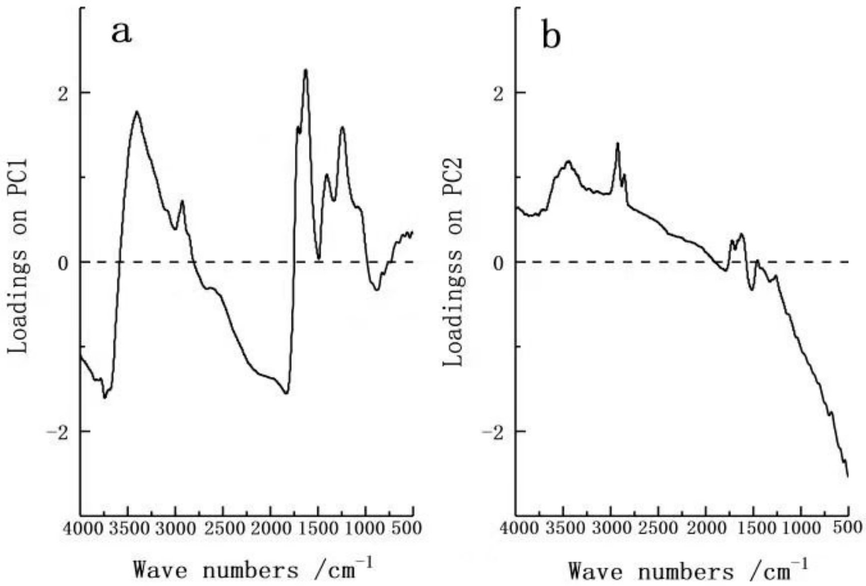

Figure 4. PCA of soil HA Infrared Spectroscopy.

3.5. *The Fluorescence Emission Spectra of HA*

From Figure 5, the fluorescence emission spectra of HA treated by different shapes of corn straws were similar in shape. The peak wavelengths were similar; however, each had a unique peak at a long wavelength. There were similar fluorophores in different treatments of HA. Comparing the CK-treated HA fluorescence peak position (580 nm) with that of GS and CS-treated HA fluorescence peak positions, the HA fluorescence peak positions for treatment GS and treatment CS were blue-shifted at 570 nm and 575 nm, respectively.

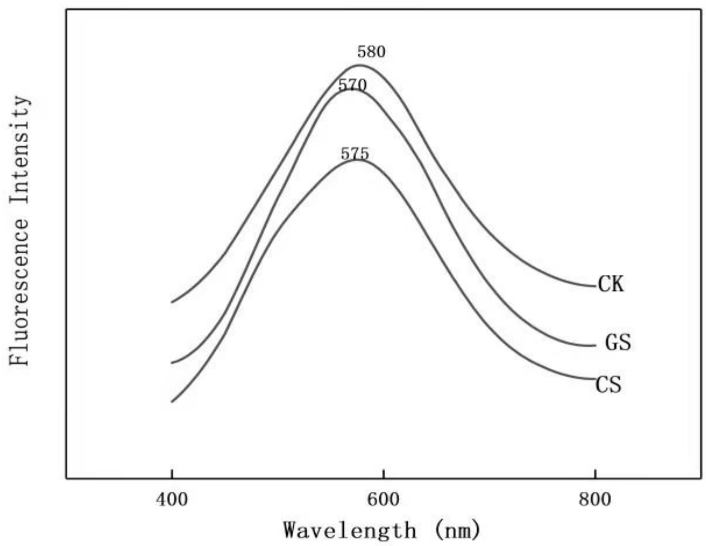

Figure 5. Fluorescence emission spectra of HA.

Table 5 shows the changes in the fluorescence intensity of HA treated with different shapes of corn straws. Comparing the fluorescence intensity of HA in treatment CK to treatment GS and treatment CS, the fluorescence intensity of HA in treatment GS and CS decreased by 14.85% and 10.24%, respectively. Comparing the HA fluorescence intensity of treatment GS with treatment CS, the HA fluorescence intensity of treatment GS was higher than treatment CS. The HA fluorescence intensity for treatment CS decreased by 5.14%.

Table 5. The intensity of peaks of HA fluorescence emission spectra.

Treatments	Fluorescence Intensity
CS	37.97
CK	42.3
GS	36.02

4. Discussion

Humus is a special form of organic matter in the soil [30] and an increase in soil humus is of great relevance to maintaining soil fertility, improving soil quality, and maintaining soil carbon pool balance. Comparing the three treatments, the treatments that involved returning straw (GS and CS) increased the soil humus and its components. This was consistent with the study conducted by Fan et al. [31]. The main reason for this observation can be attributed to a large amount of carbon source materials found in straws. After applying straw to the field, they decompose and release carbon in the form of carbon dioxide or organic carbon fixed in the soil [32]. These carbon source materials are the main source of soil humus. In addition, an increase in microbial metabolism could be a reason for the increased soil humus and its components. This is because studies have reported that the decomposition of fresh straw increases the activity and abundance of soil microbial communities, which promotes the metabolism of microbial communities and the accumulation of their products [33,34]. Soil microbial residues and their products are important carbon sinks. The amino sugars (AS) in the microbial cell wall are composed of 2%–7% carbon, which is closely related to the turnover of carbon-containing organic matter, such as soil organic matter [33]. The increase in microbial activity, in turn, promotes the decomposition of straw, and it is generally believed that fungi are more effective in the decomposition of straw [35]. This was also consistent with the findings of Liu et al. [36].

The E4/E6 ratio and ΔlogK values are optical properties that reflect the complexity of the molecular structure of soil HA [37]. The higher the E4/E6 ratio and ΔlogK value, the smaller the optical density, aromaticity, and the degree of soil HA humification. In this experiment, the application of corn straw increased the E4/E6 ratio and ΔlogK value of soil HA, which resulted in a reduction of the optical density, aromaticity, and the degree of soil HA humification. The molecular structure of HA was also reduced to a simple structure.

The functional groups on the surface of humic acid molecules in the soil have bands which are characterized by their obligatory characteristics in the infrared spectrum. The infrared spectroscopy can be used to analyze the functional group composition of soil humic acid [11]. Principal component analysis (PCA), is considered to be an important method in establishing a minimum data set. PCA mainly transforms multiple indicators into a few indicators through dimensionality reduction, which eliminates the correlation between multiple indicators and makes them independent [38]. The PCA results reveal that the application of corn straw is one of the primary sources of aliphatic carbon, aromatic carbon, and amino compounds in the soil. The results also show that the application of straw caused a reduction in aromaticity, the degree of condensation, and the degree of oxidation of soil humic acid structure. However, the aliphaticity was enhanced, and the humic acid structure appeared to be aliphatic, simple, and young. Through the analysis of the soil fluorescence spectrum, it was observed that returning straw to the field caused the blue shift phenomenon, which indicates that the degree of humification is low. The simplified molecular structure of HA is a result of the reduction in fluorescence intensity [39]. Most studies obtained similar results. Al-Faiyz [30] analyzed the structural characteristics of soil humic acid after composting with different organic materials by ^{13}C NMR. The results of this study showed that the aliphaticity and aliphatic content of humic acids were high under the corn straw treatments. These observations were directly attributed to the application of corn straw. Brunetti et al. [40] also reported a decrease in the degree of condensation of HA structure in the soil after applying organic fertilizer, however, the aliphaticity increased and the HA structure appeared to be aliphatic. The application of straw can increase the organic carbon source [41] and also contribute to the improvement of soil microbial activity [42]. Generally, the formation of humus is closely related to the activities of microorganisms. The interaction between soil microbes and stable humus can completely or partially decompose humus, produce fresh humus, and continuously renew the soil organic matter pool [43].

Comparing the two straw treatments, treatment GS increased the amount of humus and its components as compared to treatment CS. The soil humic acid structure for treatment GS appeared to be aliphatic, simple, and young. This could be attributed to the damaged surface layer of the corn straw during the pulverization process, which accelerated the transformation of the straw materials that are difficult to decompose [44]. The granular corn straw (treatment GS) was pulverized and compacted, which makes it absorb water to form a loose porous structure. This increases the contact area between microorganisms and the straw and creates better growth and development conditions for the microorganisms, which greatly promotes the microbial activity and microbial decomposition of the corn straw, producing humus [45].

5. Conclusions

The initial hypothesis of the study can be accepted since the application of straw increased the content of soil humus and its related components. This affected the humic acid structure by making it appear to be aliphatic, simple, young and also promotes the continuous renewal of humus. The relative content of aliphatic carbon in soil humic acid increased, and the relative content of the carboxyl group in soil humic acid decreased. The results show that the granular straw reduced the condensation and oxidation of humic acid structure, enhanced the aliphatic group, decreased the aromaticity, and made the humic acid structure tend to be aliphatic, simplified, and young. Therefore, it can be concluded that different shapes of corn straw have different effects on soil humus and its related components, and the effect of granular corn straw is better and highly recommended. This study also shows that corn straw can be used for the organic fertilization of barren, native saline-alkali land, to promote sustainable agricultural development.

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References

1. Li, H. Evaluation on the Production of Food Crop Straw in China from 2006 to 2014. *Bioenergy Res.* **2017**, *10*, 949–957. [CrossRef]
2. Fei, L.U.; Wang, X.; Han, B.; Ouyang, Z.; Duan, X. Soil carbon sequestrations by nitrogen fertilizer application, straw return and no-tillage in China's cropland. *Global Chang. Biol.* **2009**, *15*, 281–305.
3. Yin, H.; Zhao, W.; Li, T.; Cheng, X.; Liu, Q. Balancing straw returning and chemical fertilizers in China: Role of straw nutrient resources. *Renew. Sustain. Energy Rev.* **2017**, *81*, 2695–2702. [CrossRef]

4. Frouz, J. Effects of soil macro- and mesofauna on litter decomposition and soil organic matter stabilization. *Geoderma* **2017**, *332*, 161–172. [CrossRef]
5. Wood, S.A.; Tirfessa, D.; Baudron, F. Soil organic matter underlies crop nutritional quality and productivity in smallholder agriculture. *Agric. Ecosyst. Envrion.* **2018**, *266*, 100–108. [CrossRef]
6. Yanagi, Y.; Shindo, H. Assessment of long-term compost application on physical, chemical, and biological properties, as well as fertility, of soil in a field subjected to double cropping. *Agric. Sci.* **2016**, *07*, 30–43. [CrossRef]
7. Adani, F.; Genevini, P.; Ricca, G.; Tambone, F.; Montoneri, E. Modification of soil humic matter after 4 years of compost application. *Waste Manag.* **2007**, *27*, 319–324. [CrossRef]
8. Zhu, Q.T.; Shen, L.Y.; Qian, L.H.; Guo, S.W.; Dai, J.Y. Effects of organic material application on the structures of humic acids in low yield paddy soil. *J. Soil Sci. Plant Nut.* **2015**, *21*, 396–403.
9. Xu, D.; Zhou, X.; Wang, X. Adsorption and desorption of Ni^{2+} on Na-montmorillonite: Effect of pH, ionic strength, fulvic acid, humic acid and addition sequences. *Appl. Clay Sci.* **2008**, *39*, 133–141. [CrossRef]
10. Li, J.J.; Li, C.Z.; Gao, L.J.; Liu, B.S.; Liu, D.S.; Zou, G.Y. Impact of different organic fertilizers on soil humus, microbiomass and quality of cabbage. *Adv. Mater. Res.* **2014**, *955*, 628–632. [CrossRef]
11. Rodríguez, F.J.; Schlenger, P.; García-Valverde, M. Monitoring changes in the structure and properties of humic substances following ozonation using UV-Vis, FTIR, and 1H NMR techniques. *Sci. Total Environ.* **2016**, *541*, 623–637. [CrossRef] [PubMed]
12. Li, Q.; Sun, L.; Zhang, Y.; Qian, Y.; Zhai, J. Characteristics of equilibrium, kinetics studies for adsorption of Hg (II) and Cr (VI) by polyaniline/humic acid composite. *Desalination* **2011**, *266*, 188–194. [CrossRef]
13. Kogut, B.M.; Sysuev, S.A.; Kholodov, V.A. Water stability and labile humic substances of typical chernozems under different land use. *Eurasian Soil Sci.* **2012**, *45*, 496–502. [CrossRef]
14. Hu, J.; Wu, J.G.; Qu, X.J.; Li, J.M. Effects of organic wastes on structural characterizations of humic acid in semiarid soil under plastic mulched drip irrigation. *Chemosphere* **2018**, *200*, 313–321. [CrossRef] [PubMed]
15. Yan, C.; Yan, S.S.; Jia, T.Y.; Dong, S.K.; Ma, C.M.; Gong, Z.P. Decomposition characteristics of rice straw returned to the soil in northeast China. *Nutr. Cycl. Agroecosys.* **2019**, *114*, 211–224. [CrossRef]
16. Garnier, P.; Cambier, C.; Bousso, M.; Masse, D.; Chenu, C.; Recous, S. Modeling the influence of soil-plant residue contact on carbon mineralization: comparison of a compartmental approach and a 3d spatial approach. *Soil Biol. Biochem.* **2008**, *40*, 2754–2761. [CrossRef]
17. Angers, D.A.; Recous, S. Decomposition of wheat straw and rye residues as affected by particle size. *Plant Soil.* **1997**, *189*, 197–203. [CrossRef]
18. Yanardağ, I.H.; Zornoza, R.; Bastida, F.; Büyükkiliç-Yanardağ, A.; García, C.; Faz, A.; Mermut, A.R. Native soil organic matter conditions the response of microbial communities to organic inputs with different stability. *Geoderma* **2017**, *295*, 1–9. [CrossRef]
19. Qadir, M.; Oster, J.D.; Schubert, S.; Noble, A.D.; Sahrawat, K.L. Phytoremediation of sodic and saline-sodic soils. *Adv. Agron.* **2007**, *96*, 197–247.
20. Yang, J.; Zhang, S.; Li, Y.; Bu, K.; Zhang, Y.; Chang, L.; Zhang, Y. Dynamics of saline-alkali land and its ecological regionalization in western Songnen Plain, China. *Chin. Geogr. Sci.* **2010**, *20*, 159–166. [CrossRef]
21. Guan, S.; Dou, S.; Chen, G.; Wang, G.; Zhuang, J. Isotopic characterization of sequestration and transformation of plant residue carbon in relation to soil aggregation dynamics. *Appl. Soil Ecol.* **2015**, *96*, 18–24. [CrossRef]
22. Schollenberger, C.J. Determination of soil organic matter. *Soil Sci.* **1945**, *59*, 53–56. [CrossRef]
23. Campitelli, P.; Ceppi, S. Effects of composting technologies on the chemical and physicochemical properties of humic acids. *Geoderma* **2008**, *144*, 325–333. [CrossRef]
24. Sparks, D.L.; Helmke, P.A.; Page, A.L. *Methods of Soil Analysis: Chemical Methods*; American Society of Agronomy: Madison, WI, USA, 1996; No. 631.417/S736 V. 3.
25. Cerli, C.; Celi, L.; Kaiser, K.; Guggenberger, G.; Zanini, E. Changes in humic substances along an age sequence of Norway spruce stands planted on former agricultural land. *Org. Geochem.* **2008**, *39*, 1269–1280. [CrossRef]
26. Senesi, N.D.; Orazio, V.; Ricca, G. Humic acids in the first generation of eurosoils. *Geoderma* **2003**, *116*, 325–344. [CrossRef]
27. Zhang, J.J.; Wang, L.B.; Li, C.L. Humus characteristics after maize residues degradation in soil amended with different copper concentrations. *Plant Soil Environ.* **2011**, *56*, 120–124. [CrossRef]
28. Pechenizkiy, M.; Puuronen, S.; Tsymbal, A. The impact of sample reduction on PCA-based feature extraction for supervised learning, Acm Symposium on Applied Computing. *ACM* **2006**, 553–558. [CrossRef]

29. Cui, T.; Li, Z.; Wang, S. Effects of in-situ straw decomposition on composition of humus and structure of humic acid at different soil depths. *J. Soil Sediment.* **2017**, *17*, 2391–2399. [CrossRef]
30. Al-Faiyz, Y.S.S. CPMAS ^{13}C NMR characterization of humic acids from composted agricultural Saudi waste. *Arab. J. Chem.* **2017**, *10*, S839–S853. [CrossRef]
31. Fan, W.; Wu, J.G.; Li, J.M.; Hu, J. Comparative effects of different maize straw returning modes on soil humus composition and humic acid structural characteristics in northeast China. *Chem. Ecol.* **2018**, *34*, 355–370. [CrossRef]
32. Zhao, X.M.; He, L.; Zhang, Z.D.; Wang, H.B.; Zhao, L.P. Simulation of accumulation and mineralization (CO_2 release) of organic carbon in chernozem under different straw return ways after corn harvesting. *Soil Till. Res.* **2016**, *156*, 148–154. [CrossRef]
33. Maarastawi, S.A.; Frindte, K.; Geer, R.; Kröber, E.; Knief, C. Temporal dynamics and compartment specific rice straw degradation in bulk soil and the rhizosphere of maize. *Soil Biol. Biochem.* **2018**, *127*, 200–212. [CrossRef]
34. Blagodatskaya, E.V.; Blagodatsky, S.A.; Anderson, T.H.; Kuzyakov, Y. Contrasting effects of glucose, living roots and maize straw on microbial growth kinetics and substrate availability in soil. *Eur. J. Soil Sci.* **2009**, *60*, 186–197. [CrossRef]
35. Schneider, T.; Keiblinger, K.M.; Schmid, E.; Sterflinger-Gleixner, K.; Ellersdorfer, G.; Roschitzki, B.; Richter, A.; Eberl, L.; Zechmeister-Boltenstern, S.; Riedel, K. Who is who in litter decomposition? Metaproteomics reveals major microbial players and their biogeochemical functions. *ISME J.* **2012**, *6*, 1749. [CrossRef]
36. Liu, X.; Zhou, F.; Hu, G.; Shao, S.; He, H.; Zhang, W.; Zhang, X.; Li, L. Dynamic contribution of microbial residues to soil organic matter accumulation influenced by maize straw mulching. *Geoderma* **2019**, *333*, 35–42. [CrossRef]
37. Liu, S.; Zhang, Z.Y. Response of soil carbon dioxide fluxes, soil organic carbon and microbial biomass carbon to biochar amendment: a meta-analysis. *GCB Bioenergy* **2016**, *8*, 392–406. [CrossRef]
38. Kardanpour, Z.; Jacobsen, O.S.; Esbensen, K.H. Soil heterogeneity characterization using PCA (Xvariogram)-Multivariate analysis of spatial signatures for optimal sampling purposes. *Chemom. Intell. Lab.* **2014**, *136*, 24–35. [CrossRef]
39. Miano, T.M.; Senesi, N. Synchronous excitation fluorescence spectroscopy applied to soil humic substances chemistry. *Sci. Total Environ.* **1992**, *117*, 41–51. [CrossRef]
40. Brunetti, G.; Plaza, C.; Clapp, C.E.; Senesi, N. Compositional and functional features of humic acids from organic amendments and amended soils in Minnesota, USA. *Soil Biol. Biochem.* **2006**, *39*, 1355–1365. [CrossRef]
41. Zhang, J.J.; Cao, Z.Y.; Feng, G.Z.; Li, M.Y.; Li, C.L.; Gao, Q.; Wang, L.C. Effects of integrated soil-crop system management on soil organic carbon characteristics in a Primosol in Northeast China. *Pedosphere* **2017**, *27*, 957–967. [CrossRef]
42. Jackson, L.E.; Calderon, F.J.; Steenwerth, K.L.; Scow, K.M.; Rolston, D.E. Responses of soil microbial processes and community structure to tillage events and implications for soil quality. *Geoderma* **2003**, *114*, 305–317. [CrossRef]
43. Abiven, S.; Menasseri, S.; Chenu, C. The effects of organic inputs over time on soil aggregate stability—A literature analysis. *Soil Biol. Biochem.* **2009**, *41*, 1–12. [CrossRef]
44. Bremer, E.; Houtum, W.; Kessel, C. Carbon dioxide evolution from wheat and lentil residues as affected by grinding, added nitrogen, and the absence of soil. *Biol. Fert. Soils* **1991**, *11*, 221–227. [CrossRef]
45. Lou, Y.; Liang, W.; Xu, M.; He, X.; Wang, Y.; Zhao, K. Straw coverage alleviates seasonal variability of the topsoil microbial biomass and activity. *Catena* **2011**, *86*, 117–120. [CrossRef]