

秸秆深还对土壤腐殖质组成和胡敏酸结构特征的影响*

崔婷婷¹ 窦森^{1†} 杨轶因¹ 陈丽珍¹ 黄毅² 王瑞丽²

(1 吉林农业大学资源与环境学院, 长春 130118)

(2 沈阳农业大学土地与环境学院, 沈阳 110161)

摘要 以辽宁省阜新县试验基地培肥 3 年的草甸褐土为研究对象, 设 CK(不施秸秆)和 CS(秸秆深还施用量为 24 000 kg hm⁻²) 2 个处理, 每个处理随机选取 3 个采样点, 采集深度为 0~20 cm 和 20~40 cm。采用腐殖质组成修改法提取水溶性物质(WSS)、胡敏酸(HA)、富里酸(FA)和胡敏素(Hu), 研究秸秆深还对土壤腐殖质各组分含量的影响, 并提取土壤胡敏酸(HA)固体样品进行结构表征。研究表明: 秸秆深还更有助于表层土壤及其各腐殖质组分 WSS、HA、FA 和 Hu 有机碳含量的积累, 分别较 CK 增加了 33.11%、26.39%、11.09%、9.197% 和 18.55%, 亚表层土壤 WSS 有机碳含量变化不显著, 但 HA 和 Hu 的含碳量降低; 表层和亚表层土壤 PQ 值的变化不显著; 表层和亚表层土壤 HA, 缩合度变大, 分子结构变复杂, 芳香结构增加, 热稳定性相对提高, 亚表层土壤 HA 脂族结构稳定性增强。

关键词 秸秆深还; 土壤腐殖质组分; 胡敏酸; 元素组成; 差热分析; 红外光谱

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土壤有机质是土壤的重要组成成分^[1], 是衡量土壤肥力的重要指标^[2]。腐殖物质是土壤有机质的最重要组分之一, 它们是深色的、非均质的化合物^[3]。而胡敏酸是土壤腐殖质中的活跃物质, 其组成结构和性质的变化与土壤的保肥和供肥性质相关^[4]。在土壤急需培肥和农业机械化水平提高的情况下, 秸秆深还成为了提高土壤肥力, 增加土壤有机质的重要手段之一。万运帆等^[5]研究表明秸秆深施与表施相比, 土壤有机碳显著增加近 1%, 温室气体 N₂O 的排放量以有机肥处理最大, 秸秆深施和秸秆覆盖较小。杨振权和陈永星^[6]对水田秸秆机械深施养分释放进行了研究, 经土壤分析测定表明: 秸秆还田处理, 使 N、P、K 和 C 的全量和速效养分均显著提高; 而且秸秆深施又较面施养分增加的显著。常晓慧, 孔德刚等^[7-8]研究表明玉米、大豆和水稻秸秆深施均可以提高土壤蓄水能力, 可以减缓土壤温度下降速度, 保障了农作物发芽和成长。Børresen^[9]的研究结果表明, 施入秸秆会增加作物产量, 并且对于干旱年间作用更加明显。Bhatta-

charyya 等^[10]试验研究表明, 施入水稻秸秆 + 无机氮肥的处理可显著增加表层土壤的全碳量和碳储量。Humberto 和 Lal^[11]研究表明, 免耕-秸秆覆盖使 0~5 cm 土层的土壤团聚体的稳定性、强度、保水能力均有所提高, 使大团聚体中的碳增加, 微团聚体里的碳减少。从国内外的研究现状来看, 对于秸秆还田的研究主要是对土壤养分、作物产量及气体排放的研究, 对于秸秆深还对土壤腐殖质的含量和结构性质研究较少。所以本文研究秸秆深还对土壤表层和亚表层腐殖质组分有机碳含量的变化、以及对胡敏酸结构特征的影响, 为明确不同腐殖质组分对土壤固碳和肥力的贡献提供理论基础。

1 材料与方法

1.1 试验设计

以辽宁省阜新蒙古族自治县西扣莫村为试验基地, 试验设未施秸秆(CK)和秸秆深还(CS)2 个处理。玉米收获时采用机械深开沟, 上底宽 60 cm, 下底宽

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† 通讯作者, 窦森(1959—), 男, 教授, 博士生导师, 主要从事土壤有机质研究。E-mail: dousen1959@126.com

作者简介: 崔婷婷(1988—), 女, 硕士研究生, 从事土壤环境与生物化学研究。E-mail: 852086928@qq.com

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40 cm, 高 40 cm。开沟后秸秆亚表层深还用量为 24 000 kg hm⁻², 并配施化肥, 秋季覆膜, 翌年春季大垄双行二比空种植耐密型玉米, 具体设计参考文献 [12]。取样时间为 2012 年 10 月, 每个处理随机选取 3 个采样点, 采集深度为 0~20 cm 和 20~40 cm。

1.2 分析方法

采用腐殖质组成修改法^[13-14]提取水溶性物质(WSS)、胡敏酸(HA)、富里酸(FA)和胡敏素(Hu), 并测其有机碳含量。

HA 的提取和纯化: 将过 60 目干土 100 g 按土:液=1:10 的比例, 用 0.1 mol L⁻¹ NaOH 在室温下提取。向上述溶液中加入 2.5 mol L⁻¹ HCl 酸化至 pH=1.5, 酸化了的混合液置于低速离心管中离心, 离心管中沉淀即为粗 HA, 粗 HA 进一步处理具体步骤见文献 [15]。

土壤及胡敏素有机碳的测定均采用重铬酸钾外加热法^[16]; HA、FA 等水溶性有机碳含量采用岛津 TOC-VCPH 仪测定; 热性质分析采用日本岛津 TA-60 型差热分析仪测定, 并应用仪器自带软件分析各样品的差热分析(Differential thermal analysis, DTA)和热重分析(Thermogravimetric analysis, TGA); 红外光谱采用 KBr 压片法^[17]在美国 NICOLET-AV360 型红外光谱仪上测定, 扫描模式为 4 000~400 cm⁻¹。对谱

线选取特征峰, 并对相应的官能团进行半定量分析。

1.3 统计分析

数据经 Excel 2003 整理, 用 SPSS 软件分析, 采用 LSD 法进行显著性差异比较。差热分析图和红外光谱图使用 Origin 软件分析作图。

2 结果与讨论

2.1 秸秆深还对土壤有机碳含量的影响

秸秆深还对土壤及其腐殖质组分有机碳含量的影响见表 1。不同土层的土壤及其腐殖质组分的有机碳含量均表现为表层>亚表层。秸秆深还后, 表层土壤及其腐殖质各组分 WSS、FA、HA、Hu 的有机碳含量均有所上升, 分别增加了 33.11%、26.39%、9.197%、11.09% 和 18.55%; 而亚表层除 WSS 外, 土壤以及 HA、FA、Hu 的有机碳含量均有不同程度的降低。可见表层土壤有机碳量提高的幅度大, 说明秸秆深还有助于土壤有机碳量的积累, 秸秆还田对土壤有机碳变化起到极为重要的影响, 这与南雄雄等^[18]研究结果一致。由表 2 可以看出, 秸秆深还后表层土壤的 WSS、FA、HA 及 Hu 的相对含量均有不同程度的降低; 亚表层除 HA 外, WSS、FA 和 Hu 的相对含量均有不同程度的上升。

表 1 秸秆深还对土壤及其腐殖质组分有机碳含量的影响

Table 1 Effect of deep-applied corn stalk on organic carbon contents in the soil and various fractions of soil humus (g kg⁻¹)

处理 Treatment	采集深度 Depth (cm)	原土 Soil	水溶性物质 WSS	富里酸 FA	胡敏酸 HA	胡敏素 Hu
CK	0~20	8.061 ± 1.169b	0.072 ± 0.002b	1.435 ± 0.016b	1.884 ± 0.170ab	4.281 ± 0.829ab
	20~40	6.334 ± 0.181c	0.053 ± 0.005c	1.332 ± 0.062c	1.642 ± 0.168bc	3.181 ± 0.235bc
CS	0~20	10.73 ± 1.08a	0.091 ± 0.010a	1.567 ± 0.018a	2.093 ± 0.190a	5.075 ± 0.575a
	20~40	5.933 ± 0.296c	0.060 ± 0.001c	1.269 ± 0.040c	1.476 ± 0.193c	3.118 ± 0.638c

注: CK 为不施秸秆的对照, CS 为秸秆深还处理; WSS 为水溶性物质, FA 为富里酸, HA 为胡敏酸, Hu 为胡敏素。同一列中小写字母代表显著水平 $p < 0.05$ 。下同 Note: CK stands for control without corn stalk application; CS for treatment with corn stalk applied; WSS for water-soluble substances, FA for fulvic acid, HA for humic acid, and Hu for humin. The lower-case letters in the same column mean significant difference at level $p < 0.05$. The same below

表 2 秸秆深还对土壤腐殖质组分有机碳相对含量的影响

Table 2 Effect of deep-applied corn stalk on relative contents of the components of soil humus (%)

处理 Treatment	采集深度 Depth (cm)	水溶性物质 WSS	富里酸 FA	胡敏酸 HA	胡敏素 Hu
CK	0~20	0.90 ± 0.11ab	18.06 ± 2.63a	23.52 ± 1.58ab	52.83 ± 2.86a
	20~40	0.84 ± 0.06b	21.02 ± 0.42a	25.89 ± 2.01a	50.18 ± 2.43a
CS	0~20	0.85 ± 0.05b	14.70 ± 1.39b	19.52 ± 0.37b	47.30 ± 2.25a
	20~40	1.01 ± 0.46a	21.44 ± 1.74a	24.97 ± 3.92a	52.92 ± 12.73a

2.2 秸秆深还对 PQ 值的影响

PQ 值为 HA 在腐殖酸 (HA + FA) 中的比例,是反映有机质腐殖化程度的指标。图 1 为秸秆深还后不同耕层 PQ 值,可以看出表层 PQ 值差异不显著,从 CK 的 56.69% 至 CS 的 57.11%,变化不明显;而亚表层 PQ 值降低,从 CK 的 55.14% 降至 CS 的 53.63%。说明秸秆深还能降低土壤的腐殖化程度,使 FA 缩合成 HA 的作用减弱。这是由于随着培肥时间的增加,秸秆基本腐解,这与陈丽珍^[19]的研究的结果一致。一些长期定位试验中也发现使用有机物料会导致腐殖酸含量、PQ 值等显著变化^[20]。

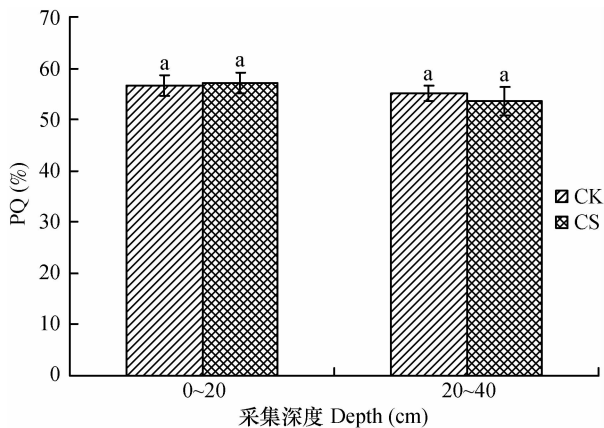


图 1 秸秆深还对 PQ 值的影响

Fig. 1 Effect of deep-applied corn stalk on PQ of the soil

2.3 秸秆深还对土壤胡敏酸元素组成的影响

元素组成分析是判断腐殖质结构和性质最简单、最重要的方法之一。通过对元素的分析,可以简单的判断腐殖质可能的组成与结构。秸秆深还

对土壤 HA 元素组成的影响列于表 3,可以看出胡敏酸的元素组成以碳为主,其次为氧。HA 的 C 含量为 540.3 ~ 591.9 g kg⁻¹,CK 处理表层 HA 的 C 含量大于亚表层,CS 处理表层 HA 的 C 含量小于亚表层;表层土壤 HA 的 C 含量,CK 处理大于 CS 处理,亚表层则为 CK 处理小于 CS 处理,表明秸秆深施有助于亚表层土壤 HA 的 C 含量积累。H 含量和 N 含量分别为 62.08 ~ 73.30 和 42.99 ~ 51.92 g kg⁻¹,CK 和 CS 处理使表层 HA 的 H 和 N 含量均小于亚表层;表层和亚表层土壤 HA 的 H 和 N 含量,CK 处理均大于 CS 处理。O 含量为 288.2 ~ 354.7 g kg⁻¹,CK 处理表层 HA 的 O 含量小于亚表层,CS 处理表层 HA 的 O 含量大于亚表层;表层土壤 HA 的 O 含量,CK 处理小于 CS 处理,亚表层则为 CK 处理大于 CS 处理。一般认为 H/C 和 O/C 是表征 HA 缩合度和氧化程度的指标。对于 O/C 来说,CK 处理表层 HA 的 O/C 小于亚表层,CS 处理表层 HA 的 O/C 大于亚表层;表层土壤 HA 的 O/C 为 CK 处理小于 CS 处理,亚表层则为 CK 处理大于 CS 处理,说明秸秆深还使表层土壤 HA 氧化度提高,亚表层 HA 氧化度下降。CK 和 CS 处理使表层 HA 的 H/C 均小于亚表层;表层和亚表层土壤 HA 的 H/C 均为 CK 处理大于 CS 处理,说明秸秆深还后土壤 HA 的缩合度变大,分子结构变复杂,芳香性提高,所以秸秆深还增加了土壤 HA 的固碳能力。有研究表明,施用稻草后,H/C 的比值逐渐降低,说明土壤 HA 的脂肪族的成分在不断增加,由于脂肪族化合物是较稳定难分解的,意味着土壤胡敏酸的稳定性在增加,土壤胡敏酸的变化是朝着更加稳定性的基团转化^[21]。

表 3 秸秆深还对土壤 HA 元素组成的影响

Table 3 Effect of deep-applied corn stalk on elemental composition of HA in the soil

处理 Treatment	采集深度 Depth (cm)	C (g kg ⁻¹)	H (g kg ⁻¹)	N (g kg ⁻¹)	O (g kg ⁻¹)	C/N	H/C	O/C
CK	0 ~ 20	589.5	71.50	50.05	289.0	13.74	1.456	0.368
	20 ~ 40	550.6	73.30	51.82	324.3	12.40	1.598	0.442
CS	0 ~ 20	540.3	62.08	42.99	354.7	14.66	1.379	0.492
	20 ~ 40	591.9	70.34	49.61	288.2	13.92	1.426	0.365

2.4 秸秆深还对土壤胡敏酸结构特征的影响

2.4.1 胡敏酸热学性质的变化

由图 2 可知,各处理 HA 的 DTA 均存在低温吸热峰 (49 ~ 59 °C)、中温放热峰 (296 ~ 336 °C) 和高温放热峰 (393 ~ 413 °C),高温放热明显高于中温放热。中温峰温以

CS 处理的表层最高,亚表层最低。由半定量积分结果(表 4)亦可知,CS 处理的表层和亚表层土壤 HA 在高温处的总反应热均大于相应的对照处理,说明秸秆深还使 HA 芳核结构增多或缩合程度提高^[22]。秸秆深还使表层和亚表层 HA 的放热高温/中温值

从 8.40 上升至 30.67 和从 5.74 上升至 20.70, 芳香结构或高温放热成分比例增加, 热稳定性相对提高。热重分析表明(表 5), 秸秆深还使表层和亚表层 HA 热失重的高温/中温值分别从 2.11 上升至

2.81、从 1.95 上升至 2.85, 也说明芳香结构或高温放热成分比例增加, 热稳定性相对提高, 与放热的高温/中温值变化规律一致。

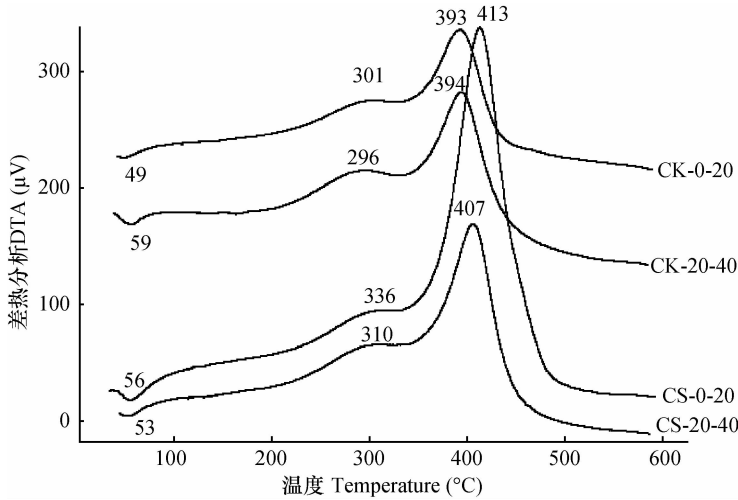


图 2 秸秆深还对土壤 HA 差热分析的影响

Fig. 2 Effect of deep-applied corn stalk on DTA of HA in the soil

表 4 秸秆深还对 HA 反应热的影响

Table 4 Effect of deep-applied corn stalk on reaction heat of HA in the soil

处理 Treatment	采集深度 Depth (cm)	低温吸热 LTE Low temperature endotherm (kJ g^{-1})	中温放热 MTE Medium temperature exotherm (kJ g^{-1})	高温放热 HTE High temperature exotherm (kJ g^{-1})	总反应热 Total reaction heat (kJ g^{-1})	高温/中温值 HTE/MTE
CK	0 ~ 20	-0.15	2.33	19.57	21.82	8.40
	20 ~ 40	-0.61	2.90	16.64	19.46	5.74
CS	0 ~ 20	-0.59	0.92	28.22	29.06	30.67
	20 ~ 40	-0.20	0.94	19.46	20.32	20.70

表 5 秸秆深还对 HA 失重的影响

Table 5 Effect of deep-applied corn stalk on weight loss of HA in the soil

处理 Treatment	采集深度 Depth (cm)	低温失重 LTWL Lowtemperature weight loss (mg g^{-1})	中温失重 MTWL Mediumtemperature weight loss (mg g^{-1})	高温失重 HTWL Hightemperature weight loss (mg g^{-1})	总失重 Total weight loss (mg g^{-1})	高温/中温值 HTWL/MTWL
CK	0 ~ 20	47.88	191.5	404.0	643.4	2.11
	20 ~ 40	78.67	185.2	360.4	624.3	1.95
CS	0 ~ 20	89.84	146.1	409.9	645.9	2.81
	20 ~ 40	66.49	137.2	391.3	594.9	2.85

2.4.2 胡敏酸红外光谱的变化 从图 3 中可以看到, HA 的 IR 图谱具有相似的特征, 说明不同处理 HA 具有基本一致的结构, 但在某些特征峰吸收强度上有不同程度的差异, 反映了不同处理对 HA

结构单元和官能团的数量有一些影响。这表明, 土壤 HA 发生的变化主要是由于秸秆 HA 和腐解形成的 HA 进入土壤所致^[23]。从图 3 中可以看出, 秸秆深还后亚表层 HA 在 2 920、2 850、1 720、1 620、

1 030 cm^{-1} 处较对照振动剧烈,说明秸秆深还后 HA 羧基的 C=O 伸缩振动作用增强,芳香 C=C 伸展振动增强,以及芳香酯中的 C-O 伸展增强,而且从表 6 可看到 2 920/1 620 值也较对照高,说明新形成

的 HA 具有较强的脂族性。秸秆深还 3 年后表层土壤 HA 除了在 2 920 cm^{-1} 处低于相应的 CK 处理外,在其他各处均大于对照。

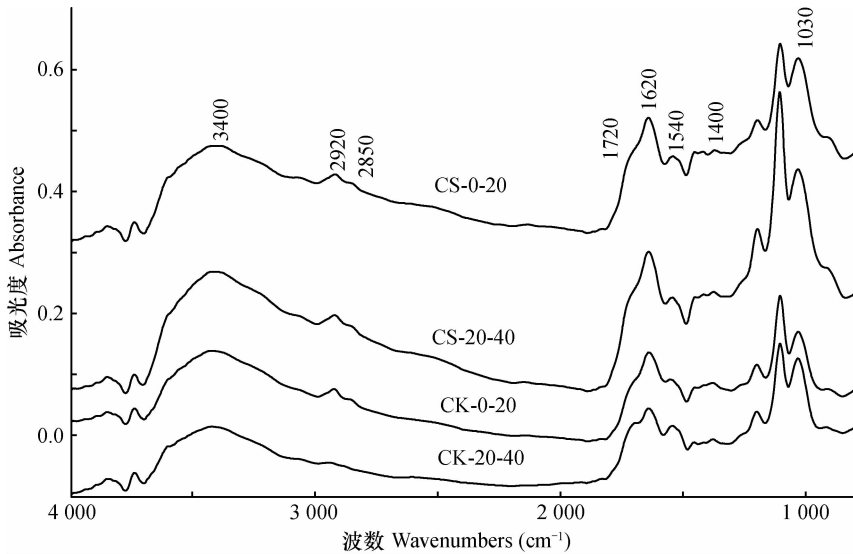


图 3 秸秆深还对土壤 HA 的 IR 光谱的影响

Fig. 3 Effect of deep-applied corn stalk on IR of HA in the soil

表 6 HA 的 IR 光谱主要吸收峰的相对强度(半定量)

Table 6 Relative intensity of the main absorption peaks in IR spectrum of HA (Semi-quantitative)

处理 Treatment	采集深度 Depth (cm)	3 400 (cm^{-1})	2 920 (cm^{-1})	2 850 (cm^{-1})	1 720 (cm^{-1})	1 620 (cm^{-1})	1 540 (cm^{-1})	1 400 (cm^{-1})	1 030 (cm^{-1})	2 920/ 2 850	2 920/ 1 720	2 920/ 1 620
CK	0~20	38.70	0.73	0.21	0.59	2.35	0.72	0.62	3.10	3.48	1.59	0.40
	20~40	31.59	0.17	0.11	0.79	1.53	1.42	—	3.94	1.55	0.35	0.18
CS	0~20	59.26	0.71	0.29	1.05	4.13	1.10	—	4.71	2.45	0.95	0.24
	20~40	45.03	0.74	0.35	0.81	2.95	1.00	—	4.41	2.11	1.35	0.37

3 结论

对于阜新地区潮褐土而言,秸秆深还 3 年后,表层土壤有机碳含量显著提高,其各腐殖质组分含碳量也有所增加,表层土壤 PQ 值没有显著变化;亚表层土壤有机碳含量变化不显著,其腐殖质组分仅 HA 及 Hu 碳量有所降低,亚表层土壤 PQ 值无显著变化。从元素分析、差热分析和红外光谱来看,秸秆深还后表层和亚表层土壤中 HA 缩合度变大,芳香结构和高温放热成分比例增加,热稳定性提高,亚表层土壤 HA 脂族性也有所提高。

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EFFECT OF DEEP APPLIED CORN STALKS ON COMPOSITION OF SOIL HUMUS AND STRUCTURE OF HUMIC ACID

Cui Tingting¹ Dou Sen^{1*} Yang Yinan¹ Chen Lizhen¹ Huang Yi² Wang Ruili²

(1 College of Resources and Environment, Jilin Agricultural University, Changchun 130118, China)

(2 College of Land and Environment, Shenyang Agricultural University, Shenyang 110161, China)

Abstract Soil, as a natural resource essential to human survival, can provide people with food and fiber they need in their daily life and production. In recent years of rapid economic development, people over-exploit the land and apply irrationally fertilizers, thus triggering a series of problems, like land degradation, reduced grain output, food security and so on, which in turn threatens sustainable development of the human society. Therefore, soil amelioration has become a problem attracting extensive concerns and discussions the world over. Corn stalk is a renewable kind of resource, rich in supply and ready for direct use. Northeast China is a major corn producing region of the country and turns out every year large volumes of corn stalk, which unfortunately is either used as cooking fuel or just burnt in the field, thus leading to a great waste of organic resource and pollution of the environment as well. In the case that the soil urgently needs fertilizing and mechanical farming improving, the practice of deep application of corn stalk is recommended to be an important measure to improve soil fertility, increase soil organic matter, and hence to promote agricultural production and guarantee people's life. Soil organic matter is a key factor of fertile farmland and humic substances are important components of organic matter and considered to be the most abundant organic ingredients of the nature, and they play a critical role in the ecosystem. Humic substances can be classified, according to their solubility, into humic acid (HA), fulvic acid (FA) and humin (Hu). HA is a kind of polymer compound, complex in structure and soluble in base solution, but not in water or acid. HA is also an important extract of humus.

From a field of Cinnamon soil fertilized for 3 years in a field experiment base inXokoumo Village, Fuxin Mongolia Autonomous County, Liaoning Province, soil samples were collected in October 2012. The field experiment was designed to have two treatments, CK (no stalk applied) and CS (stalk applied at a rate of 24 000 kg hm⁻²). Each treatment had three randomly-selected sampling points, from which soil samples were gathered at 0 ~ 20 cm and 20 ~ 40 cm for analysis in lab. Water-soluble substances (WSS), humic acid (HA), fulvic acid (FA) and humin (Hu) were extracted using the modified humus component extraction method to explore effects of deep applied corn stalk on content of organic carbon in various humus components in the soil. Solid HA was extracted from the soil as samples for characterization of its structure through elemental composition analysis, differential thermal analysis and infrared spectral analysis. Results show that deep-applied corn stalk helped increase the content of organic carbon in the surface soil and in WSS, HA, FA and Hu therein, by 33.13%, 28.57%, 11.33%, 9.57% and 18.58%, respectively. It raised, somewhat, the content of organic carbon in WSS too, but reduced the content in, HA and Hu in the subsurface soil; it lowered the relative contents of WSS, FA and HA in the surface soil to a varying extent, but did not vary much the relative contents of all of them except WSS in the subsurface soil, to a varying degree; PQ values of the surface and subsurface soils did not vary much; From the perspective of element composition analysis, it is generally held that H/C and O/C are the factors characterizing condensation and oxidation levels of HA. Compared with CK, Treatment CS was relatively lower in H/C of HA in the surface and subsurface soil, indicating that deep-applied corn stalk raises the condensation degree of HA and complicates its molecular structure, and Treatment CS was higher in O/C of HA in the surface soil but lower in the subsurface soil, indicating that the application of corn stalk intensifies HA oxidation in surface soil, but lessens the reaction in the subsurface soil; The differential thermal analysis shows that the high/medium temperature values in exotherm and weight loss of HA in the surface and subsurface soils both went up, indicating that HA increases its aromatic structure and improves its thermal stability relatively; The infrared spectrum analysis shows that deep-applied corn stalk intensified C = O stretching vi-

bration of HA carboxyl, C = C stretching vibration of aroma and C-O stretching vibration of aromatic ester, and moreover, the semi-quantity system analysis reveals that the 2920/1620 value was higher in CS than in CK, indicating that newly formed HA has strong aliphatic nature.

Key words Deep-applied corn stalk; Soil humus composition; HA; Elemental composition; Differential thermal analysis; Infrared spectrometry

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