



# Article Effects of the Ingredients on Maturity and Humification during Kitchen Waste Composting as Illustrated by Nuclear Magnetic Resonance

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**Abstract:** Kitchen waste (KW) production has risen considerably due to the increasing affluence of populations and a booming catering sector. If not properly disposed, KW can bring serious issues for the environment. Composting is widely used as an efficient method for the resource utilization of KW. In the present paper, the effects of different ratios of ingredients (corn straw: garden waste = 4:1, based on the dry mass) on maturity and humification during KW composting were investigated. The results showed that the nitrogen retention capacity of the treatments with a ratio of ingredients to KW of 1:2.5 and 1:5.0 was higher than that of other treatments. Additionally, it was found that the number of ingredients had an effect on the germination index (GI) during composting, and the final GI values of the four treatments were in the following order: 1:7.5 > 1:5.0 > 1:10.0 > 1:2.5. Nuclear magnetic resonance (NMR) showed that the organic matter (OM) content of each treatment increased after composting, and the maturity of the 1:2.5 and 1:5.0 treatments was higher than the other two treatments. Redundancy analysis (RDA) showed that the maturity and humification were correlated with moisture content, pH and NO<sub>3</sub><sup>-</sup>-N during composting. This study concludes that the treatment with an ingredients to KW ratio of 1:5.0 was much more useful for KW composting, which is of importance to guide the disposal of KW.

Keywords: kitchen waste; ingredients; composting; physicochemical property; maturity; humification

# 1. Introduction

Kitchen waste (KW) is a generic term that refers to non-edible food and leftover food from households, restaurants, and canteens, which is increasing in quantity due to rapid urbanization and population growth in recent decades [1–3]. KW includes complicated ingredients such as food remnants, oils and fats, bones, peel, waste tableware, plastic, and paper towels. KW with more than 70% of moisture content (MC) are the high organic contents of crude protein and crude fat, up to 14.45–28.86% and 17.02–29.30% [4,5]. In China, approximately 30 million tons of KW is generated each year, which accounts for approximately 50–70% of municipal solid wastes (MSW) in almost all cities, such as Beijing 66.2%, Shanghai 60.4%, Guangzhou 61.6%, Hangzhou 56.8%, Nanjing 52.8%, Shenzhen 51.1%, and Xiamen 65.3% [6–8]. When KW is exposed to the environment without treatment, in time, the leachate, foul odor, and harmful gas emissions produced by KW can cause pollution of soil and the atmosphere [9–12]. To alleviate excessive accumulation, we should pay more attention to the responsible treatment and disposal of the increasing amounts of KW with multiple organic nutrient contents.

The processing and/or disposal options for food waste, including KW, mainly include incineration, landfilling, and composting [7,13]. Incineration and sanitary landfills have



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**Copyright:** © 2023 by the authors. Licensee MDPI, Basel, Switzerland. This article is an open access article distributed under the terms and conditions of the Creative Commons Attribution (CC BY) license (https:// creativecommons.org/licenses/by/ 4.0/). been widely used for the disposal of KW, but pose more and more environmental and social problems, such as air pollution, greenhouse gas emissions, leachate generation, risk of fires, and disease spread via pests and animal scavengers [5,6,14–16]. Composting is regarded as the universal recycling of disposals, to convert KW into value-added products for sustainable management and resource recovery [3]. Due to the high proportion of biodegradable organic compounds, KW could be transformed into fertilizer for recycling and utilization as resources [17,18]. Land application after composting is a preferable and practical choice for the disposal of KW in China; Soil fertility and soil physical properties can be improved through this inexpensive and easy practice. However, the high MC in KW is a main barrier that influences  $O_2$  supply and microbial metabolism in composting [19,20]. Therefore, it is necessary to accelerate humification processes and improve maturation efficiency through the addition of the auxiliary materials in KW composting.

Several studies investigated maturity and gaseous emissions in KW composting with various ingredients. Bulking agents such as cornstalks, sawdust, and mushroom substrates were widely used for KW composting, and enhanced compost maturity and reduced greenhouse gas emissions including  $CH_4$  and  $N_2O$  emissions [21,22]. Yang et al. (2015) reported that the addition of phosphogypsum and superphosphate in KW composting had no negative effect on maturity and significantly decreased  $CH_4$  and  $NH_3$  emissions [23]. Margaritis et al. (2018) indicated that KW composting using different mineral additives such as zeolite and perlite improved the germination index (GI) in almost all seeds [24]. However, the humification processes of compost substances during KW composting with different ingredients have rarely been discussed. Nuclear magnetic resonance (NMR) spectroscopy is a promising tool to characterize the structure distribution of humic substances and track composting humification. Therefore, we selected KW and accessible ingredients (corn straw and garden waste) as compost raw materials to explore the maturity and humification of composting using different ratios of KW to ingredients via NMR. Specifically, the following hypotheses were tested: (i) compost process parameters would be affected by different ratios of mixed complementary materials and KW during composting, and (ii) the maturity and humification of composting have a certain relevance in some process parameters. The purpose of this investigation was to explore the appropriate ratio of KW and ingredients contributing to the maturity of composting, and to further provide a theoretical basis for the resource utilization of KW and other solid wastes.

#### 2. Materials and Methods

#### 2.1. Composting Materials

KW was obtained from a cafeteria in Beijing, and the composition mainly included leftovers, fruit peels, uneatable vegetables, and waste food. The ingredients, corn straw and garden waste collected from the experiment site (Langfang, Hebei, China), were mixed at a ratio of 4:1 based on the dry weight (DW) and were passed through a 0.5 cm-pore sieve after an air-drying pretreatment [25]. All the raw materials were divided into two parts. One part was preserved for the composting experiment. The other was used for detecting the selected physicochemical properties (Table 1). The wheat (*Triticum aestivum* L.) cultivar Zhongmai 12 was obtained for the GI trials.

**Table 1.** The physicochemical properties of the selected composting materials <sup>1</sup>.

Composting Materials	pН	MC (%)	TOC (%)	TN (%)	C/N
Kitchen waste	4.38	79.12	47.68	2.51	18.99
Corn straw	7.68	8.40	45.11	0.85	53.07
Garden waste	5.85	21.13	54.25	2.01	26.99

<sup>1</sup> MC, moisture content; TOC, total organic carbon; TN, total nitrogen; C/N, the ratio of TOC and TN content.

#### 2.2. Experimental Design and Implementation

According to the farmers' habits for household composting, four ratios of ingredients with kitchen waste based on the dry weight were set as 1:2.5, 1:5.0, 1:7.5, and 1:10.0 (T1,

T2, T3, and T4, respectively). Each ratio was replicated three times. A certain amount of KW and the ingredients were mixed well to get the given ratios after incomplete airing. The initial MC of the compost mixtures was adjusted as approximately 60% using distilled water. Additionally, the C/N ratio was adjusted to approximately 25:1 by urea. Then, the composting mixtures of each treatment were divided into three heaps and transferred into the foam compost reactors (inner size  $66 \times 46 \times 41$  cm, thickness 3.5 cm) for 60 days. The weight of every heap is approximately 60.0 kg. During the composting period, the MC of composting mixture was not artificially adjusted. A temperature probe was inserted into the center of each heap to record the real-time temperature during composting. To ensure adequate oxygen supply, the composting piles were manually turned on alternate days for the first 18 days of composting, and then once every week.

#### 2.3. Sample Collection and Analysis

According to the temperature profile of the composting piles, the samples were collected from the upper, middle and lower positions of the composting pile and mixed completely every ten days, on days 0, 10, 20, 30, 40, 50, and 60. To investigate the progress of decomposition and the maturation of the compost during a period of 60 days, the samples were subjected to analysis of certain physical and chemical indexes including pH, electrical conductivity (EC), the MC, ammonium nitrogen ( $NH_4^+$ -N), nitrate nitrogen ( $NO_3^-$ -N), dissolved organic carbon (DOC), the GI and spectral characteristics.

The MC (%) was calculated by finding the difference between the weights of wet and dry (dried at 105 °C) samples. The pH was determined for a slurry consisting of compost and distilled water (1:10, w/v) with an MP521 pH/EC meter (Sanxing, Shanghai, China). The DOC was determined with a TOC automatic analyzer. NH<sub>4</sub><sup>+</sup>-N and NO<sub>3</sub><sup>-</sup>-N were extracted with 2 mol L<sup>-1</sup> KCl (1:10, w/v), and then determined using the indophenol blue colorimetric method and using the phenoldisulfonic acid method, respectively [26]. The germination index (GI) was detected to assess the phytotoxicity of the compost and measured by the method described previously by Feng et al. (2021) [27].

The samples collected on days 0, 30, 60 were smashed into powder, dissolved in  $0.1 \text{ mol } \text{L}^{-1}$  NaOH solution, and then analyzed by an AVANCE III 400 MHz Digital NMR Spectrometer (Bruker, Karlsruhe, Germany). For this, 7 mm-diameter sample tubes were used. Additionally, the measurement conditions of the spectra were as follows: 100.64 MHz spectrometer frequency, 5 kHz spinning speed, 90° pulse width, 69 ms of acquisition time, and 1 s of pulse delay time. Approximately 10,000 scans were collected for the organic compounds of compost.

#### 2.4. Statistical Analysis

All the statistical analyses were conducted using Excel 2020 (Microsoft, Redmond, WA, USA) and SPSS 26.0 (IBM, Armonk, NY, USA), and the data were profiled using Origin 2023b (OriginLab, Northampton, MA, USA). Redundancy analysis was performed using Canoco 5.0 (Microcomputer Power, Ithaca, NY, USA) to verify the effects of physiochemical characteristics on the substance transformation of composting.

#### 3. Results and Discussion

#### 3.1. Changes in Temperature and Moisture Content

Temperature is a key parameter as it indicates microbial activity and the hazard potential and maturity of composting [28]; a high temperature is an effective guarantee of eliminating harmful microorganisms such as pathogenic bacteria. The experiment was carried out in the temperature-uncontrolled mode and the heat source was generated by the pile itself. The pile temperature was the intuitive performance of the composting process in Figure 1a. The starting temperature of all treatments was basically the same and slightly higher than the ambient temperature. Additionally, the temperature rose to 40 °C on day 4, and was then maintained at above 45 °C for a relative long time. The length of time that different groups took to reach the high temperature followed the order T2 > T1 > T3 > T4.

A pile temperature of over 50 °C for the four treatments was maintained for at least 10 days. Changes in the pile temperature showed a wavy curve in the latter period, and reached approximately ambient temperature after day 60. Figure 1a shows that each pile treatment appeared experienced small peaks in temperature after the high-temperature peak during the cooling period. This phenomenon may have been caused by the alternating increased metabolism of mesophilic bacteria adapting to the environment and the depletion of organic matter (OM) that was easily degraded by active microbes [22]. Compared to the other three treatments, T4 showed a slower but continuous increase in temperature in the first two weeks. The was possibly because microbial activity was impeded in T4 with a lower pH over the same period. This further caused a delay in reaching the thermophilic stage, as indicated by the relatively higher temperature of T4 compared to that of other treatments on days 24–50.



**Figure 1.** The profiles of temperature (**a**) and moisture content (**b**) during kitchen waste (KW) composting with ingredients. Error bars represent the standard deviations of triplicate measurements. T1, T2, T3, and T4 indicate that the ratio of ingredients with KW was 1:2.5, 1:5.0, 1:7.5, and 1:10.0, respectively, and this is the case hereinafter.

During the composting process, the MC is considered as an important indicator to be regulated as it influences the structure and thermal property of the compost materials, microorganism activities and OM biodegradation [29]. As shown in Figure 1b, the initial MCs in different treatments during KW composting with ingredients were 51.38% (T1), 50.67% (T2), 49.10% (T3), and 49.55% (T4), respectively. At the beginning of composting, except for treatment T4, the MCs of the treatments increased until day 10. Thereafter, all treatments showed downward trends with the composting process. At the end of the composting process, the MCs decreased to 35.67% (T1), 30.94% (T2), 17.35% (T3), and 15.41% (T4). This indicated that there was greater water loss in treatments T3 and T4 than in the other two treatments. This may be because the composting piles of a high proportion of KW contained comprehensive and abundant nutrition and plentiful aerobic microorganisms, and there was also suitable porosity. The strong respiratory and metabolic activity of the microbes improved the dissipation and utilization of moisture [24].

#### 3.2. The Evolution of pH and EC

As KW often has a high proportion of OM and a low pH level, it is necessary to add some ingredients to maintain the pH at an appropriate level and further ensure the efficiency and maturity of composting. The pH variation in the treatments with different composting materials ratios is shown in Figure 2a. Initially, pH levels in all treatments were acidic, and the addition of ingredients could weaken the acidic environment of the composting pile. In the first ten days, the pH values of three treatments except T4 showed a greater increase based on a higher initial pH, while the pH in treatment T4 was basically unchanged. This is in accordance with the opinion of Li et al. (2013), who thought the metabolic activity of aerobic microorganisms in KW is often limited due to a low pH level

in the initial stage [30]. Another study also found that adding a higher proportion of complementary materials could be conducive to conserve aerobic conditions and reduce anaerobic conditions, which leads to an acidic environment [31]. The pH of treatments T2, T3 and T4 showed a similar trend, with a rapid increment during days 20–40 of composting, and then maintained a constant alkaline environment. This occurred because organic acids began to volatilize rapidly in the thermophilic stage of composting, producing a large amount of alkaline NH<sub>3</sub> [32]. On the other hand, the pH of treatment T1 showed a dramatic increase before day 20 and an imperceptible change to reach the maximum value of 8.67 on day 30, which was significantly higher than that of other treatments (p < 0.05). After day 30, the pH of treatment T1 gradually decreased, which may have been caused by microbial nitrification [33]. In addition, large amounts of organic acids produced by microbial activity also caused a decrease in the pH during the post-composting stage [23]. According to the Pearson analysis, pH had a positive correlation with temperature (p < 0.01) and a negative correlation with the MC (p < 0.05).



Figure 2. Changes in the pH (a) and EC (b) during KW composting with ingredients.

The electrical conductivity (EC) represents the total soluble salt content in the compost, indicating whether the salt content may affect compost quality [34]. Additionally, the EC increased as complex organic molecules were gradually degraded into small molecules. KW, as one of the most important components of MSW, has relatively higher EC values compared with other wastes such as manure, straw, and sawdust [35,36]. As shown in Figure 2b, the initial EC values were in the order 2.94 (T1), 3.31 (T2), 3.49 (T3), and 3.79 (T4) mS cm<sup>-1</sup>. This indicates that EC reduction was directly related to the proportion of ingredients. Additionally, in all treatments, EC generally increased and then decreased during the whole composting process. This is likely because the complex OM was decomposed into soluble components including ammonium, phosphate, potassium, and volatile fatty acids in the earlier stage of composting [32]. Additionally, then a series of processes such as ammonia volatilization, OM stabilization and precipitation of mineral salts caused a reduction in EC [27,37]. It is generally assumed that a low EC value ( $<4 \text{ mS cm}^{-1}$ ) will not cause any toxicity to compost products for agricultural use [38,39]. The EC was 3.84 (T1), 3.34 (T2), 3.77 (T3), and 3.88 (T4) mS cm<sup>-1</sup> at the end of composting, which indicates that the products of KW composting with ingredients do not have a great effect on plant growth.

#### 3.3. Pattern of $NH_4^+$ -N and $NO_3^-$ -N Contents

Nitrogen exists in the form of  $NH_4^+-N$ ,  $NO_3^--N$  and organic nitrogen (ON) in composting. During the composting process, ON was mineralized into  $NH_4^+-N$ , and ammonia in part was easily volatilized with the increase in the pile temperature and pH, which led to the loss of nitrogen. In addition, another part of ON was transformed into  $NH_4^+-N$  and  $NO_3^--N$  and was retained in the piles, so the total contents of  $NH_4^+-N$  and  $NO_3^--N$  increased continuously with kitchen waste composting (Figure 3).



Figure 3. Variation in NH<sub>4</sub><sup>+</sup>-N (a) and NO<sub>3</sub><sup>-</sup>-N (b) during KW composting with ingredients.

Changes in NH<sub>4</sub><sup>+</sup>-N content could indicate the transformation of nitrogen during composting. As shown in Figure 3a, the NH<sub>4</sub><sup>+</sup>-N contents of each treatment increased at the beginning of the composting process, and reached a peak value in different periods. The NH<sub>4</sub><sup>+</sup>-N in different treatments increased to a maximum of 1069.84 (T1), 1045.12 (T2), 1127.05 (T3), and 1011.23 (T4) mg kg<sup>-1</sup> on days 40, 20, 30, and 30 of the composting process, respectively. This might be because the active degradation of OM and nitrogenous compounds occurred with different strengths due to the change in temperature [40,41]. Moreover, Lü et al. (2013) found that the increase in NH<sub>4</sub><sup>+</sup>-N concentration was related to the high level of urease activity that could accelerate the hydrolysis of urea [37]. In the later period of composting, the NH<sub>4</sub><sup>+</sup>-N content decreased in four treatments, which could be explained by ammonia volatilization and nitrification processes [42].

As shown in Figure 3b, the  $NO_3^{-}$ -N contents of all treatments showed increasing trends in general during the whole composting process. Thereafter, the  $NO_3^-$ -N content in treatment T1 dramatically decreased on days 30-40, which may be because the growth of chemoautotrophic nitrifying bacteria was inhibited with the increase in  $NH_4^+$ -N, thus hindering the nitrification process [31,43]. Tiquia and Tam thought that a temperature above 40 °C can inhibit the activity of nitrifying bacteria during composting [44]. However, compared with treatments T1, T3 and T4, the NO<sub>3</sub><sup>-</sup>-N content of treatment T2 had a significantly higher increase in the former 20 days of composting, especially on days 10–20. This might be because treatment T2 reached the high-temperature (>40  $^{\circ}$ C) stage earlier and microbial communities with good acclimatization for high temperatures had the capability to nitrify [45]. After day 20 of composting, the  $NO_3^-$ -N contents of all treatments showed sharp increases. The  $NO_3^-$ -N contents on day 60 were 2641.40 (T1), 2595.30 (T2), 2470.70 (T3) and 2745.80 (T4) mg kg<sup>-1</sup>, respectively. Additionally, the contents increased by 343.34% (T1), 322.48% (T2), 280.28% (T3) and 260.96% (T4), respectively, which indicated that the increasing ratio of ingredients to KW favored the accumulation of NO<sub>3</sub><sup>-</sup>-N. Cáceres et al. (2018) thought that the parameters of the raw materials such as type of feedstock and physical conditions were the main factors influencing the microbes responsible for nitrification, and that lower easily degradable carbon and the lower activity of heterotrophic bacteria would favor the conditions for nitrifying bacteria and then optimize nitrification [46].

#### 3.4. Changes in DOC Contents

DOC is an index to represent the degradable OM content dissolved or liquefied in composting materials. A high DOC content is conducive to biological fermentation of OM [47]. As shown in Figure 4, the initial DOC contents of four treatments were relatively high and presented a decreasing trend, which was basically identical to the result of Hwang et al. (2020) [36]. Treatment T4 showed the highest DOC of 11.48%, followed by treatment T3 (8.39), T2 (7.84), and T1 (7.47) at the beginning of composting. In the first 10 days, the DOC of treatments T1 and T2 increased to maximums of 7.57% and 9.76%, respectively, while

treatments T3 and T4 decreased, which might be due to the hydrolyzation of free fat into soluble fat with the microbial extracellular enzyme and the conversion of macromolecular insoluble OM into small-molecule soluble OM. This was related to the pile temperature (Figure 1a). Labile OMs such as carbohydrates and fats were rapidly biodegraded by active microorganisms under rising temperatures [48]. However, the OM was progressively degraded by the continuous growth of microorganisms, and small-molecule soluble OM was utilized by microbes and transformed into novel active substances by microorganisms, which led to the gradual decline of DOC content [49].



Figure 4. Changes in DOC contents during KW composting with ingredients.

### 3.5. The Germination Index (GI)

The GI values of all treatments experienced an obvious increase during the whole composting process (Figure 5), indicating that phytotoxic substances such as short-chain volatile fatty acids were decomposed during composting [50]. Some research on manure compost showed that there would be a slight decrease in the GI during the early phase due to the generation of ammonia and low-molecular-weight short-chain volatile fatty acids [28,50,51]. However, the GIs of four treatments in this study were lowest at the begin of composting, which might be because KW with a low pH produced less short-chain volatile fatty acids and ammonia in comparison to manure waste during composting [52]. At the end of the composting process, only treatment T3 reached a GI value above 80%, and the GIs of the other three treatments were 64.13% (T1), 78.24% (T2), and 75.13% (T4). The Pearson correlation analysis showed that the GI exhibited significantly positive correlations with temperature (r = 0.595, p < 0.05), pH (r = 0.768, p < 0.01), and NO<sub>3</sub><sup>-</sup>-N (r = 0.972, p < 0.01), but a significantly negative correlation with the MC (r = -0.907, p < 0.01). Although all treatments met the requirement of a non-toxic compost (GI  $\geq$  50%), moderate amounts of ingredients could accelerate the maturity. This would guide farmers to compost scientifically using KW and agricultural wastes, which can realize waste recycling and provide fertilizer for production.

## 3.6. <sup>13</sup>C-NMR Spectral Characteristics

The humic substance formed in the composting process is the most critical factor deciding the agricultural application of compost. The humic structures during composting are usually characterized by chemical and spectroscopic techniques. In the present study, <sup>13</sup>C-NMR was used to determine the distribution of carbon in the chemical groups in different periods of composting. Based on the various distribution ranges of carbon chemical shifts, the integrated areas of the spectra for the specific structural unit shown in Figure 6 were 0–50 ppm (alkyl carbon), 50–110 ppm (alkoxy carbon), 110–160 ppm

(aromatic carbon), 160–190 ppm (carboxyl carbon), and 190–215 ppm (carbonyl carbon). The<sup>13</sup>C-NMR assignments of the main resonance areas were illustrated in Table 2.



Figure 5. Changes in the germination index (GI) during KW composting with ingredients.



**Figure 6.** <sup>13</sup>C nuclear magnetic resonance (NMR) spectra of the samples on day 0 (**a**), day 30 (**b**), and day 60 (**c**) of KW composting. The five functional groups corresponding to different chemical shifts (ppm) include alkyl C (0–50 ppm), alkoxy C (50–110 ppm), aromatic C (110–160 ppm), carboxyl C (160–190 ppm), and carbonyl C (190–215 ppm).

Signal (ppm)	Attributed Chemical Components *		
0–50	Paraffinic carbons in alkyl chains		
50-110	Aliphatic carbons substituted by oxygen and nitrogen		
110–130	Olefinic carbons; unsubstituted aromatic carbons		
130–145	Carbon-substituted aromatic carbons		
145–160	Oxygen or nitrogen substituted aromatic carbons		
160–190	Carboxyl carbons, ester or amide carbons		
190–215	Carbonyl carbons		

**Table 2.** Assignment of resonance signals in <sup>13</sup>C NMR spectra and attributed chemical components containing organic C.

\* The interpretations are based on the data of numerous studies [53-55].

As shown in Figure 6, the signals between 10 and 40 ppm were attributable to paraffinic C in alkyl chains. In this region, the main NMR peaks at approximately 30 ppm had been assigned to polyethylene C in lipids and lipid polymers [56]. Pacchiano et al. (1993) found that the chemical shifts and properties in the aliphatic region of the spectra are consistent with that in purified cutin [57], which supports a great contribution from lipid and lipid polyesters in kitchen and garden wastes even at later composting stages [58]. Additionally, a small peak at 21 ppm is usually attributed to the acetyl groups of hemicellulose [59]. The aliphatic C replaced by oxygen and nitrogen is generally observed in the region of 50–110 ppm. The remarkable signal at 73 ppm and the following peak at approximately 105 ppm were mainly produced by carbohydrates. This phenomenon was also noted in the research of Amir et al. (2004) and Sachs et al. (2002) [53,60]. Amir et al. (2004) found that carbohydrates appear frequently at approximately 70–90, 102, 106 and 111.5 ppm [53]. In addition, the weak resonance at approximately 130 ppm is mainly produced by aromatic C, and the prominent signal at approximately 174 ppm may be derived from phenolate, carboxyl, ester and/or amide groups [61].

The integrated area of <sup>13</sup>C NMR (Table 3) showed that, during the whole period of composting, the highest content was alkoxy C, followed by alkyl C, carboxyl C, aromatic C, and carbonyl C. Additionally, aliphatic C (including alkyl C and alkoxy C) decreased with a relative increase in aromatic C. Compared with other treatments, treatment T2 showed a significant increase in the alkyl C area, and a significant decrease in the alkoxy C region. Alkoxy C, the substituted aliphatic C, usually has a high resonance, and mainly arises from carbohydrate-type structures such as polysaccharides at approximately 73 ppm. Thus, the latter structures in treatment T2 might be more easily decomposed by microbes. Due to the greater proportion of KW, carbohydrates in treatments T3 and T4 had strong resistances to microbial degradation compared with other alkyl groups. In general, the aliphatic character remained after 60 days of composting. Only a small decrease in aliphatic C took place, which might be due to the significant contribution of methoxyl groups in aromatic ethers and their persistence in microbial degradation [53]. The humification of OM during composting is related to the increasing aromatic characteristics [62]. In this study, the aromaticities of all treatments had increasing trends along with composting, while the aromaticities in treatments T1 and T2 were higher than in treatments T3 and T4 at each time. Compared with the initial aromatic C content of each treatment, T1, T2, T3 and T4 aromatic C contents increased by 43.20%, 24.44%, 62.82% and 209.92% at the end of composting. The carboxyl C contents (160–190 ppm) in treatments T1, T3 and T4 decreased in the first 30 days of composting and then increased a little after day 30, which indicated that macromolecular OM such as lipids including carboxyl C and amide C in the raw materials was degraded into low-molecular-weight substances in the thermophilic phase [56], and ammonia was volatilized due to the conversion of organic N into inorganic N [27]. On the other hand, treatment T2 showed an adverse trend, which might be related to the variation tendency of alkoxy C. Castaldi et al. (2005) found that with a progressive transformation of the polysaccharides to other oxygenated compounds, the aliphatic C

Treatments	Time	Alkyl C 0–50 ppm	Alkoxy C 50–110 ppm	Aliphatic C 0–110 ppm	Aromatic C 110–160 ppm	Carboxyl C 160–190 ppm	Carbonyl C 190–220 ppm	Aromaticity <sup>1</sup> (%)
T1	day 0	19.83	64.37	84.20	5.74	7.88	2.21	6.38
	day 30	23.45	59.58	83.03	9.04	6.48	1.45	9.82
	day 60	19.83	63.04	82.87	8.22	7.25	1.66	9.02
T2	day 0	18.46	67.95	86.41	6.75	5.69	1.16	7.25
	day 30	32.71	52.72	85.43	5.51	8.26	0.79	6.06
	day 60	23.05	58.62	81.67	8.40	7.60	2.33	9.33
T3	day 0	29.88	53.78	83.66	3.55	11.06	1.74	4.07
	day 30	25.12	59.00	84.12	5.24	9.33	1.32	5.86
	day 60	28.30	54.40	82.70	5.78	9.37	2.16	6.53
T4	day 0	23.95	62.39	86.34	2.42	9.98	1.24	2.73
	day 30	20.00	64.46	84.46	4.93	8.81	1.81	5.52
	day 60	21.36	59.20	80.56	7.50	8.84	3.11	8.52

reduced, especially carboxylic and ester groups, and aromatic structures increased during composting, indicating the partial formation of new humus polymers [63].

Table 3. Intensity distributions in <sup>13</sup>C NMR spectra of the respective composting process.

<sup>1</sup> Aromaticity = C% (110–160 ppm)/C% (0–160 ppm). T1, T2, T3, and T4 indicate that the ratio of ingredients to KW was 1:2.5, 1:5.0, 1:7.5, and 1:10.0, respectively.

# 3.7. The Relationship between Physicochemical Characteristics and Substance Transformation during Composting

Redundancy analysis (RDA) was used to evaluate the effects of physiochemical characteristics on the substance transformation of composting and the correlation between them (Figure 7), and variations in the occurrence of physiochemical properties could explain 90.82% of the variance in the transformation of OM in the treatments. Based on the Pearson correlation analysis, we investigated the relationships between physiochemical conditions and substance transformation during composting. The results found that physiochemical characteristics such as the MC, pH,  $NO_3^{-}$ -N, and DOC were the primary factors influencing substance transformation. Alkyl C and alkoxy C had no significant relationship with all the determined physiochemical characteristics. This was inconsistent with the study of Feng et al. (2021), in which the transformation of alkyl C and alkoxy C was influenced by pH [27]. This might depend on the initial pH of different raw materials; and the acidic raw materials in this study inhibited the activity of microbials and had no significant effect on the distribution of the functional groups, especially the inner distribution of aliphatic C. However, the changes in aliphatic C showed a positive correlation with the MC (r = 0.718, p < 0.01) and negative correlations with NO<sub>3</sub><sup>-</sup>-N (r = -0.783, p < 0.01) and the GI (r = -0.777, p < 0.01). In addition, aromatic C and aromaticity were significantly positively related to pH (r = 0.660 and 0.671) and NO<sub>3</sub><sup>-</sup>-N (r = 0.579 and 0.610), and adversely related to DOC (r = -0.676 and -0.672). This suggested that due to more aromatic structures, the composting products are more stable and less reactive than the raw materials chemically and biologically. This result was consistent with previous studies [54,64,65]. In conclusion, to achieve better composting effectiveness, parameters such as the MC and pH could be adjusted in the preparation period of KW composting.



**Figure 7.** Redundancy analysis (RDA) of the relationship between the main physicochemical conditions and the distribution of chemical components containing organic C during KW composting with the ingredients. The circle, square, star and triangle symbols denote the data obtained from the four treatments, T1, T2, T3, and T4. Red, green, and yellow denote the data collected on days 0, 30, and 60, respectively.

#### 4. Conclusions

Co-composting of KW with mixtures of corn straw and garden waste was an effective way to deal with the issue of KW. KW and mixtures with a ratio of 5:1 achieved better maturity and humification after 60 days of aerobic composting. NMR spectra showed higher aromaticity in the lower ratio treatments. Overall, maturity was correlated with temperature, the MC, pH and  $NO_3^-$ -N during composting, while humification was associated with an increase in aromatic C, and a decrease in aliphatic C was associated with the MC, pH and  $NO_3^-$ -N. However, further optimizations need to be conducted to increase the maturity and humification efficiency of KW containing abundant organic substances during the composting process.

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