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Study on Effects of Hydrothermal Heat Treatment Method on Fertilizer Efficiency of Biomass Waste

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Abstract: This study explores a new way of utilizing biomass waste as fertilizer based on the hydrothermal heat treatment method. Hydrothermal heat treatment was carried out on common biomass waste, including corn stalks, livestock manure, and vegetable waste. by heating them to 120°C and then to 240°C respectively, while measuring the contents of organic matter, total nutrients, and humus in hydrothermal solid products. The experimental results are as follows. With the increase in temperature, the pH of three kinds of biomass waste hydrothermal products is reduced to different degrees. The content of organic matter in the solid products of corn stalk increased with the increase of temperature, the content of organic matter in the hydrothermal products of vegetable waste was opposite to that of corn stalk, and the content of organic matter in the hydrothermal solid products of pig manure was higher than 30% at different temperatures. The total nutrient content of the hydrothermal product of corn stalk decreased first and then increased with the increase of temperature, and reached the minimum value of 10.12 g/L at 180°C. The total nutrient content of the solid product of vegetable waste showed a negative correlation with temperature, and the total nutrient content of the raw material of pig manure was 60.55 g/kg. The total nutrient content of solid products after different hydrothermal temperature treatments was 41.8–59.03 g/kg, which was lower than that of raw materials. With the increase of temperature, the humic acid content of the solid product of corn stalk first increased and then decreased, the humic acid content of hydrothermal product of pig manure continued to increase with the increase of temperature, and the humic acid content of hydrothermal product of vegetable continued to increase with the increase of temperature. At 210°C, the contents of humic acid and fulvic acid reached the maximum, and the humification rate reached the maximum of 0.856.

Keywords: Biomass; Hydrothermal technology; Organic matter; Humic acid; pH

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1. Introduction

The extensive use of chemical fertilizers and various fertilizers in recent years has increased crop production but has also brought serious damage to the environment and various soil problems [1]. The search for green pollution-free fertilizers has become a hot spot in today's society [2].

Biomass energy is a kind of renewable energy that is widely found in daily life. For example, straw, livestock, and poultry manure are biomass energy. Straw is rich in cellulose, hemicellulose, and lignin, so ways to efficiently separate and use these active ingredients have been the focus of research in recent years [3–5]. Straw has seasonal, short storage time, and perishable characteristics, and is limited by economic level and technology. A large part of the straw produced is not properly utilized, resulting in environmental pollution and waste of resources [6]. Livestock manure is a biomass commonly used in daily life. Livestock manure that has not been treated will cause serious pollution to the atmosphere, water, and soil [7–9]. Animal husbandry and livestock breeding pollution have become one of the serious environmental pollution problems facing the country [10–12]. Hence, this biomass energy should be used properly to improve the environment and bring certain economic benefits.

Hydrothermal technology has the advantages of short cycle, high efficiency, simple process, and no secondary pollution, and is an environmentally friendly treatment process, which has been widely researched globally [13–15]. In this paper, the method of hydrothermal treatment will be used to pretreat the above types of biomass and measure the change in the nutrient content of the product after treatment, mainly including organic matter, total nutrients, humus, and so on, to provide a theoretical basis for the fertilizer utilization of biomass energy.

2. Test materials and methods

2.1. Test materials

The corn stalk used in the test was taken from Zhumadian City, Henan Province, China. After harvesting, the corn stalk with good growth was selected, the whole stalk was cut off and dried, and the impurities were sorted and removed. The material was crushed with a grinder to a particle size of less than 20 mm, and the material was put into a sealed bag and stored in a refrigerator at 4°C. The corn stalk composition analysis is shown in **Table 1**.

Fresh pig manure was collected from a small private pig farm in Jiaozuo City, Henan Province. The moisture content of fresh pig manure was 75% and it was sealed in a sealed bucket and stored in a freezer. The basic properties of the tested pig manure are shown in **Table 2**.

Vegetable waste was taken from a vegetable market near Henan Polytechnic University, Jiaozuo City, Henan Province, and its dry base characteristics are shown in **Table 2**. According to the waste production coefficient of different kinds of vegetables, the mass ratio of raw materials is as follows: leaf vegetables account for about 50%, melons fruits account for about 30%, and roots and stems account for about 20%. The material is broken through the crusher to a particle size of 0.5–2 mm, and the material is fully mixed for use.

Table 1. Chemical composition analysis of corn stalk

Dextran (%)	Pentosan (%)	Acid-soluble lignin (%)	Total lignin (%)	Ash (%)	Benzenol extract (%)
38.40	22.60	2.40	22.70	6.60	3.60

Table 2. Basic properties of pig manure and vegetable waste tested

Samples	pH	Organic matter content (g/kg)	Humus content (g/kg)	Humification rate (%)	Total nitrogen content (g/kg)	Total phosphorus content (g/kg)	Total potassium content (g/kg)	Total nutrient content (g/kg)
Pig manure	6.61	498.00	158.00	0.18	39.63	15.73	5.19	60.55
Vegetable	5.63	82.20	129.86	0.17	4.03	0.43	3.62	8.08

2.2. Experimental methods

The experiment was repeated with different treatment temperatures. 30 g corn straw and 500 mL pure water, 350 g pig manure and 200 mL pure water, are mixed evenly respectively with a stirring rate of 120 r/min. The different mixtures are then heated to 120°C, 150°C, 180°C, 210°C and 240°C respectively. The vegetable waste was hydrothermally treated with the rotational speed at 150 rpm and with the reaction temperature of 120°C, 150°C, 180°C, 210°C and 240°C. The time of each reaction treatment was 30 minutes and each has 3 repeated tests. After the reaction was completed, it was cooled to room temperature naturally, the products were removed for filtration, the solid products were put into the oven for drying at 105°C, and then saved for use.

2.3. Experimental equipment

The reactor used in this experiment was purchased from Xi'an OuST Instrument Technology Co., Ltd. OST series with a volume of 1000 mL triple reactor to ensure the repeatability of the experiment. It is mainly composed of an electric heating system, reactor body, and stirring system, using electric heating method. As a comprehensive reactor, it can achieve the physical and chemical reaction of heating, stirring, cooling, and low and high-speed mixing of materials. The maximum working temperature of the reactor is 300°C, and the maximum working pressure is 8 MPa. The reactor is equipped with magnetic stirring and mechanical stirring. To ensure the full reaction of the material during the experiment, an external mechanical stirring device is used, and the speed can be controlled at 0–1200 rpm (**Figure 1**).

Figure 1. Hydrothermal reaction device

2.4. Data analysis

The data was organized, analyzed, and plotted using Excel 2019 and Origin 2018.

3. Results and discussion

3.1. Product pH analysis

Figure 2 shows the pH changes of the hydrothermal solid products of corn stalks, vegetable waste, and pig manure with temperature. It can be seen from the figure that with the increase in temperature, the pH of the hydrothermal solid products of corn straw and vegetable waste showed an obvious downward trend, and the trend change of the two was similar. At 120°C–180°C, the pH of the product decreased significantly, which may be due to the hydrolysis of hemicellulose, carbohydrate, and easily degradable protein side chain groups in the raw material in the preliminary stage of the reaction to form some acidic groups, such as carboxyl, aldehyde, and phenolic hydroxyl groups [16]. With the increase in temperature, the pH decline trend of the product slows down at 180°C–210°C. The reason is that with the increase in reaction temperature, some macromolecular substances are degraded into monomers, and small molecular organic acids are generated at the same time, resulting in a continuous decline in pH. As can be seen from **Figure 2**, the acidity of vegetable hydrothermal products is stronger than that of corn straw hydrothermal products, and the difference in acidity between the two becomes more and more obvious with the increase in temperature, indicating that more acidic groups and organic acids will be produced in the process of vegetable waste hydrothermal treatment.

The pH value of the hydrothermal products of pig manure at different temperatures is between 5.67 and 6.67, and the pH value of the hydrothermal products decreases significantly between 0°C and 180°C, which may be caused by organic acids contained in biomass. The inorganic substances and acidic substances contained on the surface of the hydrothermal solid products dissolve during the reaction process, which will also lead to a decrease in pH. Between 180°C and 240°C, the pH of the solid product has risen, which may be due to the reduction of organic acid on the surface of the hydrothermal solid product in the process of hydrothermal reaction, resulting in the alkaline enhancement of the solid product.

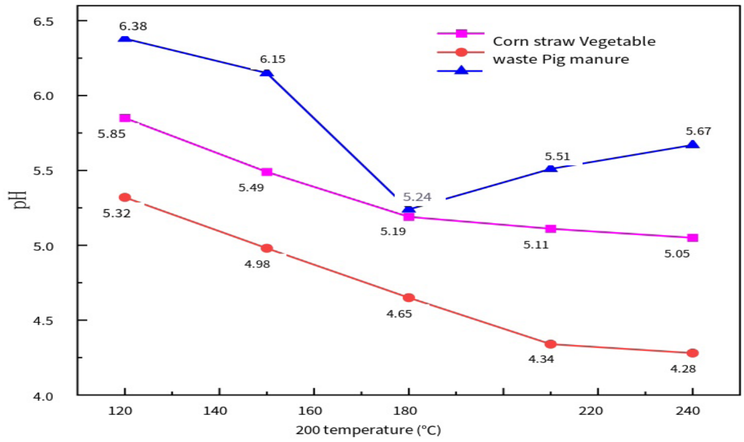

Figure 2. Change of pH of biomass hydrothermal products with temperature

3.2. Change of organic matter with temperature

Figure 3 shows the change in organic matter content of hydrothermal solid products of corn straw, vegetable waste, and pig manure with temperature. It can be seen from the figure that with the increase in temperature, the organic matter content of hydrothermal products of corn straw showed an obvious increasing trend. Compared with 120°C, the content of organic matter at 240°C increased by 77.08%, and the increasing trend was obvious from 120°C to 150°C. The organic matter content of the hydrothermal product of pig manure showed an increasing trend with increasing temperature, which was similar to that of the hydrothermal product of corn stalk, and reached the maximum value at 240°C, reaching 785.95 g/kg, which was 57.6% higher than that of raw material organic matter content of pig manure. With the progress of the hydrothermal reaction, some water-soluble small molecular organic substances, such as amino acids and sugars, were dehydrated and condensed with small molecular fragments of lignin to produce water-insoluble organic mates-humic acid, which led to the increase of organic matter content in hydrothermal products of corn straw and pig manure [17–18].

The organic matter content of the solid products of vegetable waste decreases first and then increases with the increase in temperature. At 120–180°C, the organic matter content of the product presents a negative correlation with temperature. At this stage, the easily degradable organic matter such as hemicellulose, protein, and sugar in vegetable waste is degraded into soluble sugars, monosaccharides, organic acids aldehydes, and other small organic molecules in the liquid state. Thus, the content of organic matter in the solid products decreased. After reaching 180°C, with the increase in temperature, the content of solid organic matter gradually increases. At this stage, the substances in the liquid are transformed into dissoluble substances. The substances in the liquid, such as amino acids, sugars, and aldehydes, are synthesized with lignin and other aromatic compounds to generate macromolecular organic matter that is insoluble in the liquid, such as humus substances.

There are many indexes for evaluating organic fertilizer products, with organic matter being an important index for evaluating organic fertilizer products [19–21]. It is stipulated in Organic Fertilizer (NY525-2021) that the content of organic matter in organic fertilizer should be greater than or equal to 30% [22]. It can be seen from the figure that the content of organic matter in hydrothermal products of pig manure at different temperatures is all higher than 30%. Among them, the organic matter content of the solid products at 180°C, 210°C, and 240°C is much higher than 50% of the raw materials, and the organic matter content of the solid products through hydrothermal reaction at these three temperatures has been greatly improved. The organic matter content of corn straw and vegetable waste at each treatment temperature is higher than 30%, and the organic matter content of the hydrothermal product of corn straw at 240°C is 88%, far more than 30% in the regulation.

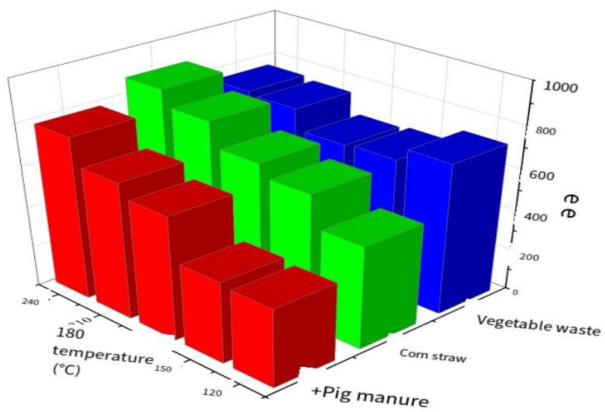

Figure 3. Changes of organic matter in solid products with temperature

3.3. Changes in total nutrients with temperature

Nitrogen, phosphorus, and potassium are the main indicators of hydrothermal product nutrients, with the total nutrients being important indicators to measure fertilizer [23–25]. **Figure 4** shows the relationship between the total nutrients of hydrothermal solid products of corn stalks, vegetable waste, and pig manure with the temperature. It can be seen from the figure that the total nutrients of hydrothermal solid products of corn stalks decrease first and then increase with the increase in temperature. In the initial stage of the reaction, hydrothermal treatment will destroy the macromolecular substances in the corn stalk and lead to its degradation. The nutrient elements in the liquid phase flow into the liquid resulting in the decrease of the total nutrient of the solid product. With the increase in temperature, the small molecular substances in the liquid product polymerize and precipitate, increasing the total nutrients of the solid product.

The total nutrient content of vegetable waste hydrothermal solid products showed a negative correlation with temperature, with the lowest total nutrient content accounting for 5.13%, which decreased by 2.5% and lost 30.9% compared with vegetable waste raw materials. Hydrothermal treatment will destroy the structure of the macromolecular substances in the vegetable waste and degrade it, resulting in the continuous loss of nutrient elements in the biomass into the liquid phase, so the total nutrient concentration in the solid product decreases with the increase of temperature.

The total nutrient content in the hydrothermal solid products of pig manure decreased with the increase in temperature, showing a negative correlation. Among them, the total nutrient content was the highest at 120°C, which was 59.03 g/kg, which decreased by 0.15% relative to the raw material of pig manure. The reason for the decrease of total nutrient content in hydrothermal solid products may be that hydrothermal heat treatment will destroy the material structure of macromolecules in pig manure, resulting in a degradation reaction. Nutrients in biomass will precipitate from the solid phase to the liquid phase, and ultimately lead to the decrease of total nutrient content in solid products. It is found that the content of nitrogen, phosphorus, and potassium in hydrothermal products of pig manure is much higher than that of corn straw and vegetable waste, which is also the reason why people use pig manure directly as fertilizer compost.

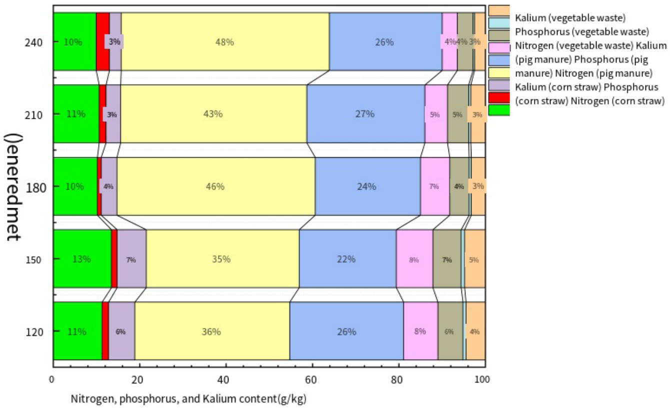

Figure 4. Relationship of total nutrients with temperature

3.4. Influence of reaction temperature on the humification process

Figure 5 shows the relationship between the humic acid of the hydrothermal solid product of corn stalk, pig manure, and vegetable waste with temperature. It can be seen from the figure that with the increase in temperature, the content of humic acid of the hydrothermal solid product of corn stalk presents an overall rising trend, and the content reaches the highest at 210°C and increases the fastest at 150°C to 180°C. This indicates that this temperature is the best temperature for the synthesis of soluble humic acid, and humic acid content tends to be stable between 180°C and 240°C. As can be seen from **Figure 6**, the humification rate of the hydrothermal product of corn stalk reached 1.42 at 210°C, which can be considered to be completely decomposed.

The humus content in the solid product of pig manure treated by hydrothermal reaction showed an increasing trend compared with the raw material, indicating that hydrothermal treatment promoted the synthesis of humus. With the increase of hydrothermal treatment temperature, the humus content in the solid product increased slowly within the temperature range of 120°C–150°C. When the temperature reached 180°C, the humus content in the solid product increased slowly. At this time, the humus content reached the highest value. As the temperature continued to rise, the content of humic acid tended to be stable, but the content of fulvic acid still increased, indicating that the conversion of fulvic acid continued at this temperature, and the organic matter was slowly transforming into humic acid.

As can be seen from the trend chart of solid humus content and humification rate of vegetable waste with temperature, humus content in solid products increases first and then decreases with increasing temperature. At 210°C, the contents of humic acid and fulvic acid reach the maximum value, and the humification rate reaches the maximum value of 0.856. The products of hydrothermal treatment are similar to those of compost. According to the standard of compost products, the products can be considered to be decomposed if the humification rate reaches 0.8 [26–27].

Compared with the hydrothermal products of the three biomass wastes, they all reach the maximum value at 210°C. The humic acid content of the hydrothermal products of vegetables is the highest, which can reach 246.5 g/kg, 174% and 137% higher than that of corn straw and pig manure, respectively. The humic acid content of hydrothermal products of corn straw and pig manure is similar, but the humification rate of hydrothermal products of corn straw is much higher than that of hydrothermal products of pig manure, and the hydrothermal products of pig manure are not completely decomposed. In addition, the variation trend of humic acid content of the three hydrothermal products was similar, all of them increased with the increase of temperature, reached the maximum value at 210°C, and then tended to be stable.

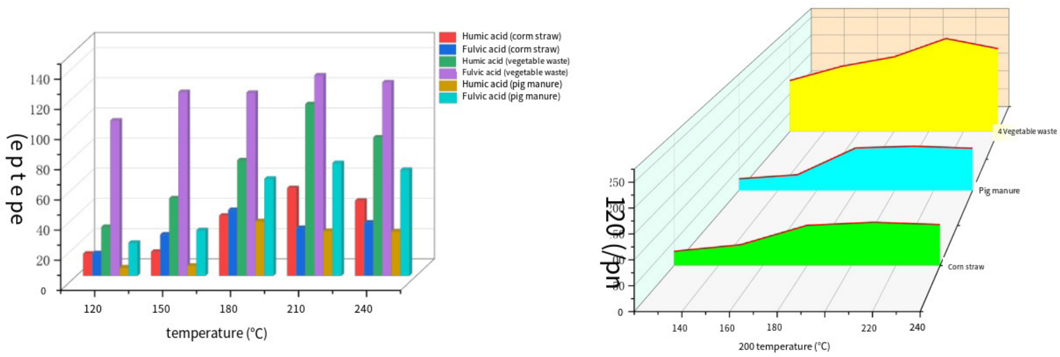

Figure 5. Changes of humic acid of biomass hydrothermal products with temperature

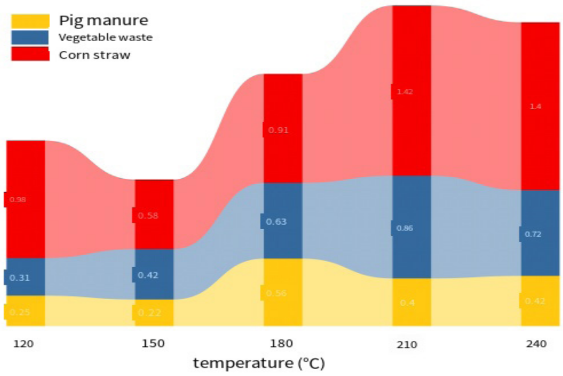

Figure 6. Changes in humification rate with temperature

4. Conclusion

This paper studied the influence of the hydrothermal method on the fertilizer of biomass waste consisting of corn stalk, livestock manure, and vegetable waste, and found that the hydrothermal method can effectively improve the fertilizer efficiency of biomass waste. The specific fertilizer efficiency index research results are as follows.

With the increase in reaction temperature, the pH of the hydrothermal solid products of corn straw and vegetable waste showed a significant downward trend, among which the acidity of vegetable waste was the strongest. The pH value of the hydrothermal solid products of pig manure was 5.67–6.67 under different hydrothermal treatment temperatures. With the increase in temperature, the content of organic matter in the hydrothermal products of corn straw showed an obvious increasing trend. Compared with 120°C, the content of organic matter increased by 77.08% at 240°C, and the content of organic matter in hydrothermal products of pig manure also showed an increasing trend with the increase of temperature, which was similar to the trend of hydrothermal products of corn straw, and reached the maximum value at 240°C, reaching 785.95 g/kg, which was 57.6% higher than the content of organic matter in raw materials of pig manure. The organic matter content of vegetable waste solid products showed a trend of first decreasing and then increasing with increasing temperature and reached the minimum value of 652 g/kg at 180°C. With the increase in reaction temperature, the total nutrients of the hydrothermal products of corn stalks, vegetable waste, and pig manure decreased continuously.

The humic acid content of the product was analyzed, and it was found that with the increase in temperature, the humic acid content of the hydrothermal solid product of corn straw showed an overall rising trend, and the content reached the highest at 210°C. The humus content of the solid product of pig manure after hydrothermal reaction treatment showed an increasing trend compared with that of the raw material, indicating that hydrothermal heat treatment had a promoting effect on the synthesis of humus. The humic acid content of vegetable waste hydrothermal products showed a trend of first increasing and then decreasing with the increase in temperature, and the humification rate reached 0.86 at 180°C, indicating it was fully decomposed.

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Disclosure statement

The authors declare no conflict of interest.

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