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Experimental Study on Carbonation Treatment

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***Abstract*—The scope of Chinese medicine residue and utilization is relatively narrow, and the harmless processing technology needs to be systematized. Since its feasibility is still being questioned by people, this research discusses the properties of the Chinese medicine residue carbonation characteristics and designs the carbide single factor test and orthogonal test by combining with the Chinese medicine residue's carbonation characteristics. In this way, the most appropriate carbonation processing parameters are determined by the element analysis of raw materials and carbonation products, combined with the reduction rate and carbonation rate before and after carbonation. Therefore, the experimental results suggest that the most appropriate carbonation processing parameters occur at the carbonation temperature of 400℃ when carbonation lasts 120 min.**

Keywords-Chinese medicine residue; carbonation treatment; carbonation rate

I. INTRODUCTION

The traditional Chinese medicine has a long history. With the progress of science and technology, the traditional Chinese medicine resources have been tapped and utilized extensively, thus the rapid development of this trade. However, the residue discharge following the traditional Chinese medicine extraction and processing becomes a big problem for numerous medicine factories, despite the huge economic benefits brought in, because many Chinese medicine manufacturing enterprises have been discharging a large amount of residue daily[1, 2]. According to statistics, a total of 30 million tons of residue are discharged annually[3].

In the past, the medical residue is usually treated by land filling, incineration and composting treatment [4], which not only has limited processing amount, but also causes huge waste of resources. Worse still, it tends to pollute environment and poses serious threats to environmental protection, logistics, security, etc. [5]. Currently, with the technology advances and people's growing awareness of medicine production specifications, the relevant enterprises and institutions have urgent demands for residue's harmless disposal and systematized use. In this case, this dissertation aims to provide evidence for making plans to make Chinese medicine residue harmless and energized, based on the study on the Chinese medicine residue's carbide characteristics and the experiment designed by considering its carbide characteristics.

II. TEST METHODS AND MATERIALS

A. *Experimental Materials and Instruments*

Experimental materials: Chinese medicine residue is extracted from the effective components of the ginkgo leaves from Shanghai Pharmaceutical Factory.

Experimental apparatus: electronic balance (FA2004N style, d = 0.001g; JM - A20002 style, d = 0.01g); electric thermostatic blast oven; muffle furnace; porcelain ark; thermogravimetric analyzer (STA409 thermogravimetric analyzer manufactured by the Germany NETZSCH Company); elemental analyzer (elemental analyzer manufactured by the Germany Elementar Vario EL CUBE).

B. *Experiment Design*

1) Thermogravimetric analysis: Thermogravimetric analysis is a way to obtain the weightlessness differential curve and integral curve of the weightless samples by discomposing them in the inert gas, based on the difference in the thermostability of various functional groups in the samples.

Chinese medicine residue, as the subject of carbonation processing, it is an important preparatory work to analyze its pyrolysis characteristics before carbonization.

Since the residue from different factories has different carbonation characteristics, the thermogravimetric analysis is an important method to study the kinetics of carbonization, considering residue's carbonation characteristics features mechanisms. The working mechanism is to make the heating furnace increase its temperature or stay at a constant one at a certain heating rate and with a temperature curve via procedure control. The experimental samples produce reaction at the procedure controlled temperature, detect and record their changes in weight and heat through sensors. Thus, with the analysis of the relationship between those data and the program temperature rise curve and temperature change, the thermogravimetric curve (TG), differential thermogravimetric curve (DTA) differential thermal curve (DTA), etc. are produced. In order to optimize residue's carbonization test, this dissertation adopts the thermogravimetric analysis method to study the law of residue weight changes at various temperatures, so as to provide sufficient evidence for the design of the residue carbonation experiment.

2) The Determination of carbonation rate and elemental analysis: Carbonization rate refers to the percentage carbon takes up in the sample weight. Thus, it not only shows the carbonation effectiveness, but also residue's actual utilization rate [6]. Based on that, this dissertation designs the single-factor experiment to investigate how carbonation temperatures affect the carbonation rate within a certain carbonization period. Experimental steps: weighing residue 20g with a constant-weight porcelain ark, accurate to 0.01g, putting the weighed samples in the muffle furnace, and setting the temperature at 300℃, 400℃ and 500℃ respectively and the carbonization time for 1.5 h for all samples. Next, taking the residue out after carbonation and cooling it down in the desiccators to the room temperature.

Elemental analysis is a quantitative analysis of specific elements for known samples. By the elemental analysis of the carbonation products and Chinese medicine residue raw materials in the above single-factor experiment, the content of C, H and N is determined by elemental analyzer from the German Elementary Vario EL Cube. Later, during the analysis and detection process, parallel detection is performed. Last, quality control is adopted on the aminobenzene sulfonic acid standard samples, and it shows the consistence between the standard samples' analytic results and actual value with the parallel errors between samples less than 3%.

3) The determination of weight loss rate: Weight loss rate refers to the percentage of samples' lost weight after processing of their original weight [7].

Reducing samples' weight and volume is the basis and precondition for residue's treatment and integral utilization, which not only improves residues treatment efficiency, but also greatly reduces the transportation costs with enhanced economic benefits. Therefore, the orthogonal experiment of two factors and three levels is designed to explore the impacts of carbonation time and carbonation temperature on residue's weight loss rate [8]. The orthogonal test table is shown in Table I below.

TABLE I. TABLE OF ORTHOGONAL TEST

Level	Carbonization Temperature/℃	Carbonization Time/min
1	300	60
2	400	90
3	500	120

III. RESULTS AND ANALYSIS

A. Thermogravimetric Analysis of Chinese Medicine Residue

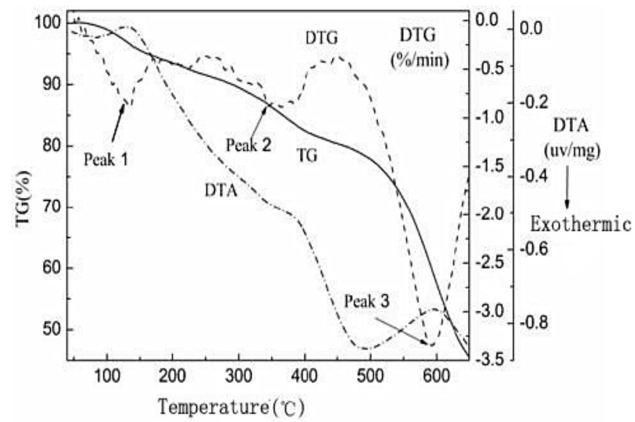

Figure 1. Curve of Chinese Medicine Residue

The DTG curve of Chinese medicine residue has three obvious peaks, showing three stages of the carbonation process. Accordingly, in the three weightlessness peaks, the DTA curve becomes an endothermic peak upward, indicating the decomposition reaction or the precipitation of volatile substances. The weightless peak occurs at the temperature of 140℃ at the first stage as an endothermic peak. Since the volatilization and precipitation of surface water and chemically combined water are an endothermic reaction, surface water and chemically combined water evaporates and volatile organic substances precipitate at low temperature. The weightless peak occurs at the temperature of 370℃ at the second stage with wide breadth, when the protein and carbohydrate in the residue decompose and mainly produce CO2, water vapor and other hydrocarbons. The third peak occurs at the temperature of 590℃, sharp. At this stage, the difficult- to- decompose organic substances decompose and those produced at the second stage begins the secondary cracking. Above 600℃, hydrocarbons, like CO2, water vapor, carboxylic acid and methane are generated, and part of carbonate decomposes. The DTG curve shows that the total weight loss at the first and second stage is 20% and 35% at the third stage when the weight loss rate is the biggest.

B. Effect of Heating Temperature and Time on Carbonization Rate and Reduction Rate

The elemental composition of N, C and H in the Chinese medicine residue can be used to evaluate its characteristics: H / C characterize aromaticity. And the lower the aromaticity, the higher the aromaticity[7] ; the content of C not only indicates the carbonation effectiveness, but also the actual utilization rate of Chinese medicine residue . The higher the content of C element, the better carbonation efficiency. As can be seen from Table II, among carbonized products, the content of H element decreases, N element increases, C element rises significantly and aromaticity correspondingly grows significantly. The content of N element in carbonized products does not vary greatly at different temperatures. For instance, the content of C element in carbonized products at 400℃ and 500℃ are similar, but slightly higher than those at 300℃. In summary, from the perspective of content of carton in carbonized products and energy conservation, carbonating at 400℃ not only has high carbon content, but also saves energy.

TABLE II. ELEMENT CONTENTS OF CHINESE MEDICINE RESIDUE

the Sample	Percentage of elements		
	C	H	N
Dregs of raw materials	44.73	6.24	0.98
The carbonization products at 300℃	58.20	4.59	1.52
The carbonization products at 400℃	59.68	3.72	1.45
The carbonization products at 500℃	59.73	3.38	1.42

Table III the weight reduction rate of the orthogonal test shows that with the carbonation time gradually increasing, residue's carbonization reduction rate rises accordingly, although at a smaller speed. With the carbonation temperature rising, residue's carbonization reduction rate grows gradually, but it only shows slight difference when at 400℃ and 500℃. When at 500℃, carbonizating consumes much energy which is uneconomic. Conclusively, residue's most appropriate carbonation parameter is the carbonation temperature at 400℃ and carbonation time of 120 min.

TABLE III. REDUCTION RATE TABLE

Carbonization Temperature/℃	300	300	300	400	400	400	500	500	500
Carbonization Time/min	120	90	60	120	90	60	120	90	60
Reduction rate/%	58.1	52.3	41.7	62.4	54.5	43.1	62.8	55.7	45.6

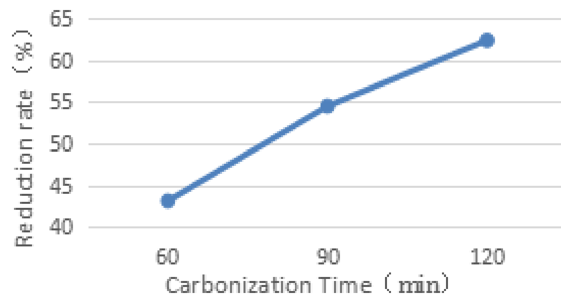

Figure 2. The reduction rate of carbonation at different times

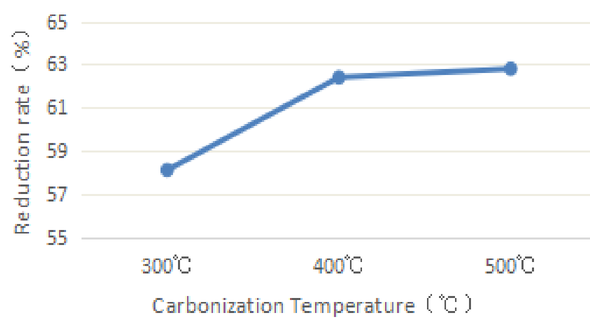

Figure 3. The reduction rate of carbonation at different temperatures

IV. CONCLUSION

In this paper, the chemical properties of traditional Chinese medicine residue have been studied by using Chinese medicine residue from Shanghai Pharma Group as raw materials, although the single factor test and orthogonal test of carbonization are designed. By analyzing the elements of raw materials and carbonized products combined with the mass reduction rate and carbonization rate before and after carbonization, it is concluded that the most appropriate processing parameters for carbonating are the carbonation temperature at 400℃ and having a time period of 120 min .In this way, energy saving is achieved, and it is also observed that carbonization products have high reduction and carbonization rate at the same time. Thus, this study is of great theoretical and practical significance to realize the reduction and harmless treatment of traditional Chinese medicine residues, resources regeneration and the sustainable development of environment.

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