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Yoshitaka Kubojima · Youki Suzuki

Measurement of the refining degree of bamboo charcoal by an alternating current method

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Abstract We investigated a method for measuring the refining degree of bamboo charcoal using an alternating current. The bamboo charcoal was made under heating conditions of 400°–900°C (set temperature) and 0–3 h (holding time at each set temperature). The qualities of the bamboo charcoal could not be estimated from the yield, and electric tests were required. The effect of the variation in sample thickness on the impedance could be ignored. Attaching two plate electrodes to the same surface of a specimen enabled high accuracy and practical use. The impedance was found to be a suitable index for estimating the refining degree of the bamboo charcoal, such as the specific electric resistance. We believe that bamboo changes from an insulator to a conductor suddenly when processed at 600°–750°C for 0–2 h. It is possible that the integral of temperature with time in a specimen during heating is useful for approximately estimating impedance.

Key words Alternating current · Bamboo charcoal · Impedance · Temperature · Time · Yield

Introduction

A charcoal refining meter has been developed as an instrument for quickly evaluating the properties of charcoal.[1] The degree of carbonization can be ascertained from the electric resistance on the surface of the charcoal using this method.

Y. Kubojima (✉) · Y. Suzuki
Forestry and Forest Products Research Institute, 1 Matsunosato, Tsukuba, Ibaraki 305-8687, Japan
Tel. +81-29-829-8302; Fax +81-29-874-3720
e-mail: kubojima@ffpri.affrc.go.jp

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The charcoal refining meter makes use of the following electrical properties of charcoal. Charcoal made at a temperature of 400°–500°C has a very high specific electric resistance of 10^8–10^5 Ωcm and is not conductive. However, if the heating temperature is higher than 800°C, the specific electric resistance of charcoal decreases to about 10^{-2} Ωcm, and this charcoal is a semiconductor. At even higher temperatures, the specific electric resistance decreases and charcoal becomes a good conductor. In the temperature range 500°–900°C that is used for making charcoal, the electric resistance is said to depend sensitively on the wood sample. There has been a great deal of research conducted on the electric resistance of charcoals made from wood and other materials.[2–4] We propose a new measuring method that uses an alternating current to estimate the refining degree of charcoal. This should be beneficial for the reasons explained below.

There are many small charcoal factories in mountain villages in Japan. In the charcoal kilns used there, temperatures tend to depend on the position of the bamboo in the kiln. Hence, the quality of the resultant bamboo charcoal is uneven. To add value to the bamboo charcoal, it should be classified according to certain factors. In the factories, the classification should be easy to perform in a short time without damaging the bamboo charcoal.

Moreover, bamboo charcoal made at lower temperatures tends to be brittle, whereas the bamboo charcoal made at higher temperatures tends to be curved. For such specimens, a plate electrode is suitable because measurements can be undertaken simply and nondestructively: the electrode can adhere to a curved surface such as a log.[5] The plate electrode is popular for dielectric measurement, for which an alternating current is used. Another example of measurements using alternating current is the high-frequency moisture meter.

However, needle-type electrodes have been used for many charcoal refining meters. Unfortunately, such electrodes will seriously damage a brittle specimen and the measurements will not be as consistent as they are for curved specimens. Examining these points, it is possible that using an alternating current could form the basis of a convenient measurement method for the factories mentioned above.

Using an alternating current may have another advantage when obtaining properties of bamboo charcoal. Various frequencies can be used for alternating currents and it is possible to pick out various parts of the structure by varying the frequency. In addition, alternating current circuits can contain condensers and coils as well as electric resistances. Therefore, we are hoping to extract more information with the alternating current than with the direct current. Although the dielectric relaxation of wood charcoal has been studied,[6–9] there have not been many results using solid samples.

Before investigating the structure inside bamboo charcoal, it is necessary to establish a measuring method with an alternating current. In this study, an alternating current and several types of electrodes were used to analyze bamboo charcoal to examine the method's effectiveness.

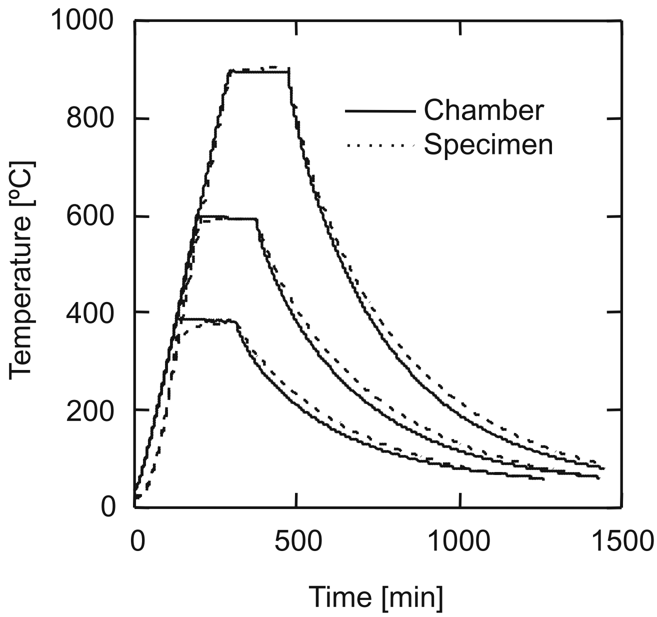

Fig. 1. Temperature changes in the oven and specimens with time (400°C and 3 h, 600°C and 3 h, 900°C and 3 h)

Experiment

Specimens

Mosochiku bamboo (*Phyllostachys pubescens* Masel, 4–5 years old) from Kagoshima Prefecture in Japan was used as the source of the specimens. Specimens with dimensions of 20–30 mm (T, tangential) in width, 10–15 mm (R, radial) in thickness (thickness of each internode), and about 200 mm (L, longitudinal) in length were made from the internodes. The thickness was measured before and after carbonization.

Carbonization

A small electric oven (Koyo Thermo Systems, KBF668N-S) was used for heating. Specimens were oven-dried at 105°C so that the experiments would not be influenced by moisture content. Thermocouples (Type K) for the measurement of the interior temperature of the specimens were set at a depth of about 30 mm from the cross section.

The set temperatures for carbonization were 400°, 500°, 600°, 650°, 700°, 750°, 800°, and 900°C and the holding times at each set temperature were 0, 0.5, 1, 2, and 3 h. The rate of temperature increase was 3°C/min. All specimens were cooled slowly after heating. During this process, nitrogen gas was circulated at a rate of 2–4 L/min through the oven. Finally the weight and electric properties of the charcoal were measured.

Figure 1 shows temperatures in the oven and of the specimens over time during heating. Examples for the set temperatures of 400°, 600°, and 900°C are shown. Temperatures showed smooth changes. The differences between the temperatures for the oven and the sample were small. The electric oven was of sufficient quality to control the temperature.

Measurement of impedance

Impedance was measured at 20°C and 65% relative humidity at 10 kHz with an LCR meter (Hioki E.E. Corporation, 3532-50 LCR HiTESTER). The 10-kHz setting was used because the most stable measurements could be obtained according to our preliminary experiments (unpublished data). The impedance is the ratio of the impressed voltage to the output of current in an alternating current circuit and shows the effect that disturbs the alternating current.

Plate and needle electrodes were employed and the results were compared. Two methods for attaching the plate electrodes to the specimens were tried. In this study, we will refer to them as "same-surface" measurements and "sandwich" measurements.

For same-surface measurements, stainless-steel plates electrodes 10 mm wide, 1 mm thick, and 50 mm long were attached to a pedestal on which a Teflon sheet was bonded. The depth of the electric field was thought to be half the distance between the electrodes.[10] The distance between the electrodes was 10 mm. The specimens were curved, making it difficult for the electrodes to come into complete contact with a specimen, and as a result, stable measurements were hard to obtain. Hence, two electrodes were brought into contact with one surface of the specimen. An insulating rubber sheet was then placed on the specimen and a 2 kg weight was placed on the rubber sheet to improve the contact (Fig. 2a). The impressed voltage was 3 V.

For the sandwich measurement, the specimen was placed between two stainless-steel electrodes. As with the same-surface measurement, contact was improved by placing a 2 kg weight on the rubber sheet (Fig. 2b). The impressed voltage was 3 V.

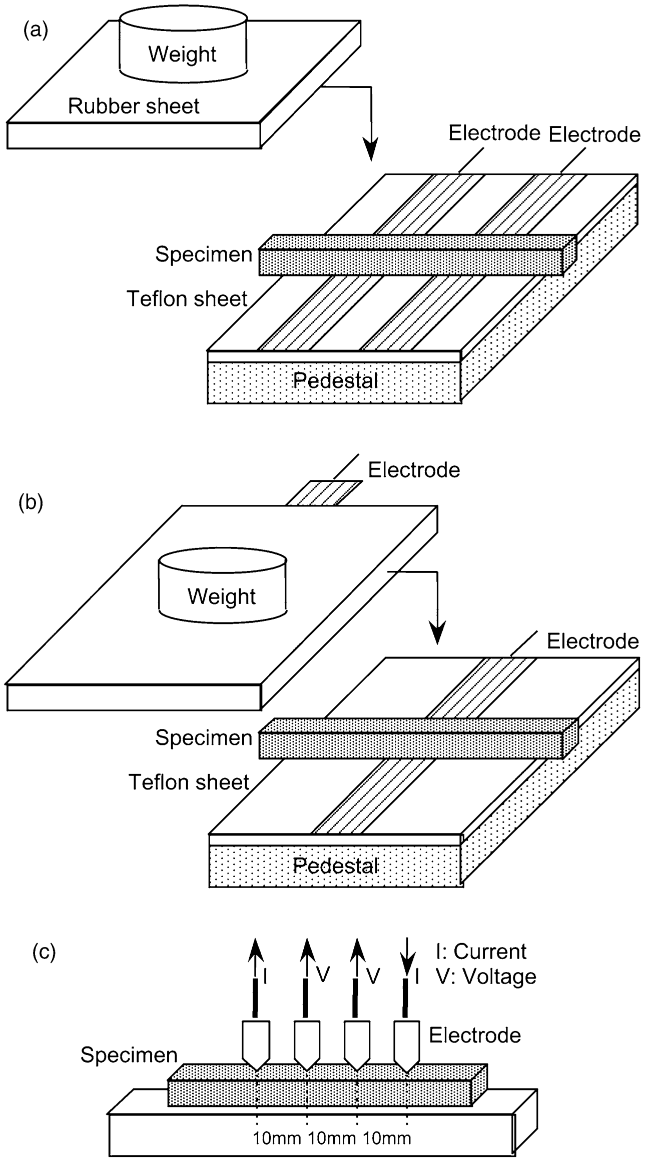

Fig. 2. Electric testing methods for bamboo charcoal: **a** same-surface measurement, **b** sandwich measurement, **c** measurement with needle electrodes

Measurement using needle electrodes followed Japanese Industrial Standard (JIS) K7194.[11] Four electrodes were used. The distance between adjoining electrodes was 10 mm (Fig. 2c) and the impressed voltage was 3 V.

Results and discussion

Yield

Figure 3 shows the yield of bamboo charcoal. The yield is the ratio of the weight directly after taking the specimens out of the oven to the oven-dried weight before carbonization. The yields were 29.3%–46.6% and more than the 13.8%–27.1% of the charcoal made in a large charcoal kiln carbonized in air[12] because of oxygen in the large charcoal kiln. The yield decreased rapidly with an increase in the set temperature from 400°–600°C, but the changes in the yield were small in the 650°–900°C range. On the other hand, the changes in the yield were small (less than 5%) for almost all holding times except at the set temperature of 400°C.

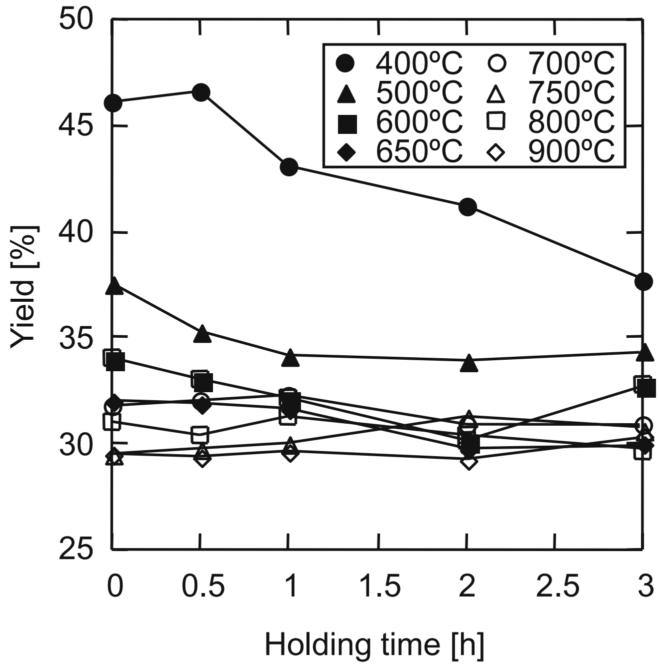

Fig. 3. Yield of bamboo charcoal at different temperatures and holding times

Effect of thickness of specimens on impedance

The effects of heating conditions on impedance are shown in Fig. 4. For the sandwich measurement, the impedance was influenced by the distance between the electrodes, i.e., the thickness of the specimen. The changes in impedance were as much as 10^6 while the average, standard deviation, and coefficient of variation of the thickness of the bamboo charcoal were 6.94 mm, 0.49 mm, and 11.33%, respectively, and so the effect of the variation in the thickness on the impedance can be ignored. This means that the change in the impedance can be attributed to the change in the conductive component of the bamboo charcoal.

Comparison of measuring methods for impedance

Several results from the needle electrodes were clearly different to those from the other two testing methods, such as the results from 650°C at 1 h and 700°C at 0.5 h. This is because of the difficulty in applying the same load to each needle electrode; some specimens were so brittle that they

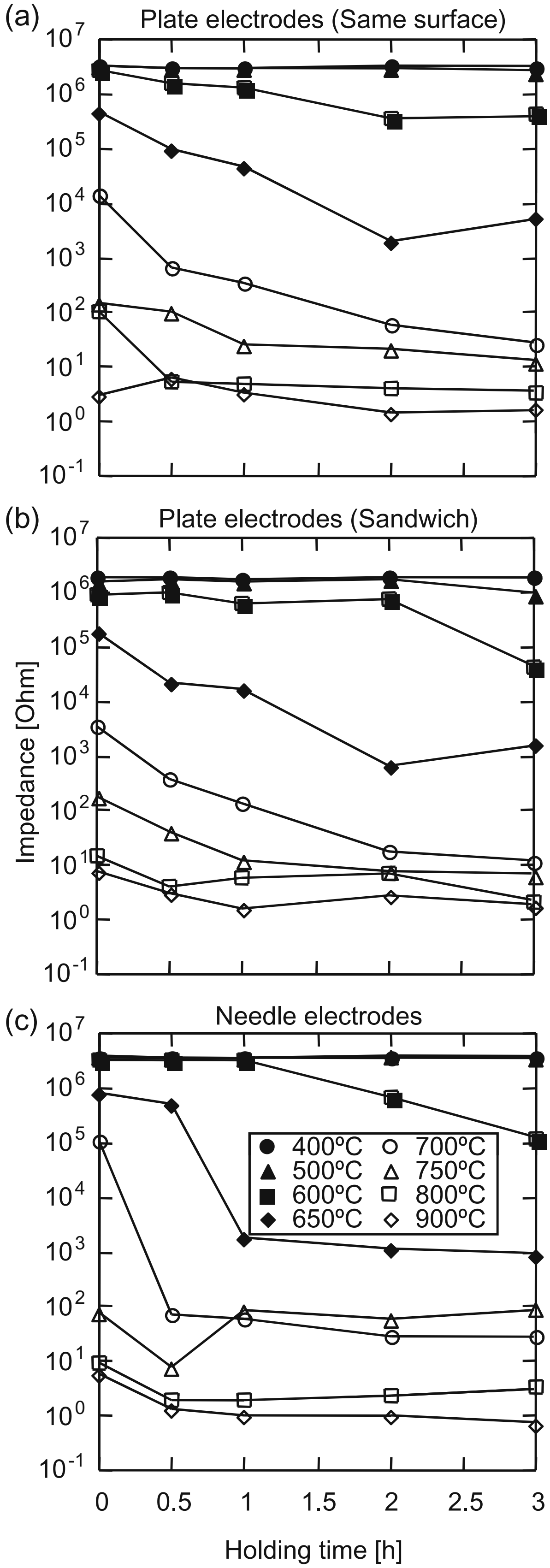

Fig. 4. Effects of heating conditions and measurement technique on the impedance of bamboo charcoal: **a** same-surface measurement, **b** sandwich measurement, **c** measurement with needle electrodes

were seriously damaged by the needle electrodes. To prevent damage, the same small load must be applied to each electrode. This requires either excellent skills or special apparatus. Furthermore, since each electrode came into contact with a specimen at one point only, measurements were not consistent. Therefore, the results obtained using needle electrodes are not reliable in the case of bamboo charcoal.

Although the total results in the thickness direction could be obtained by the sandwich measurement, clip electrodes were indispensable for measuring many specimens. The accuracy of the data can be improved by increasing the area of the electrodes. Large clip electrodes are difficult to prepare; therefore, the most practical of the three testing methods is the same-surface measurement because reliable data can be obtained simply by bringing the electrodes into contact with one surface of the specimen.

Effects of heating conditions on the impedance of the bamboo charcoal

Next, let us examine the results of the same-surface measurement and the sandwich measurement in Fig. 4. The impedance remained high for the set temperatures of 400°C and 500°C. It slightly decreased for the set temperatures of 600°C and 750°C. It decreased and was constant after 2 h for the set temperatures of 650°C and 700°C. The decrease in impedance was largest for 650°C and 700°C. Impedance was constant at low values after holding times of 0 h and 0.5 h at 800°C and 900°C. The results showed that the impedance was constant after decreasing and the constant values were lower for the higher set temperatures.

These results mean that the decrease in impedance was sensitive to heating conditions at 600°–750°C and 0–2 h. In other words, it appears that bamboo abruptly changes from an insulator to a conductor under these conditions. A similar tendency has been reported for the results of a charcoal refining meter,[4] as shown in Fig. 5. Moreover, both impedance in this study and electric resistance in a charcoal refining meter play a similar role of disturbing an electric current in a circuit. Therefore, impedance is a suitable index for estimating the refining degree of bamboo charcoal, such as the specific electric resistance. Similar tendencies for samples of Japanese cedar (*Cryptomeria japonica* D. Don) and Japanese cypress (*Chamaecyparis obtusa* Sieb. et Zucc.) in the temperature range 600°–700°C have been reported.[13]

The use of an alternating current will enable measurement of additional properties of bamboo charcoal because various frequencies can be used for alternating currents and because alternating current circuits can contain condensers and coils as well as electric resistances. The frequency dependence of electric properties is derived from various structures in the charcoal, which respond differently to lower frequencies and higher frequencies. For example, it is possible that the capacitance reflects micropores and the specific surface area in the bamboo charcoal. The impedance is expressed by various combinations of electric resistances, condensers, and coils. An electric model of bamboo charcoal using electric resistances, condensers, and coils

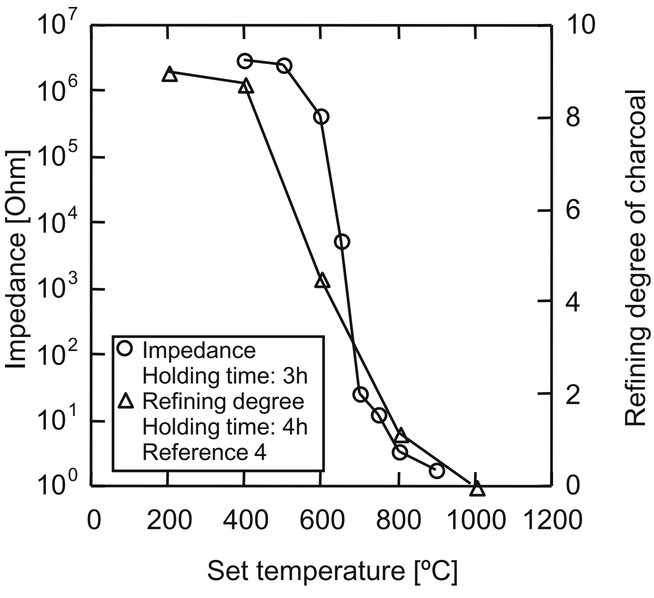

Fig. 5. Impedance and refining degree of bamboo charcoal. Refining degree is defined as the degree of carbonization expressed by the exponent of the electric resistance of charcoal[1]

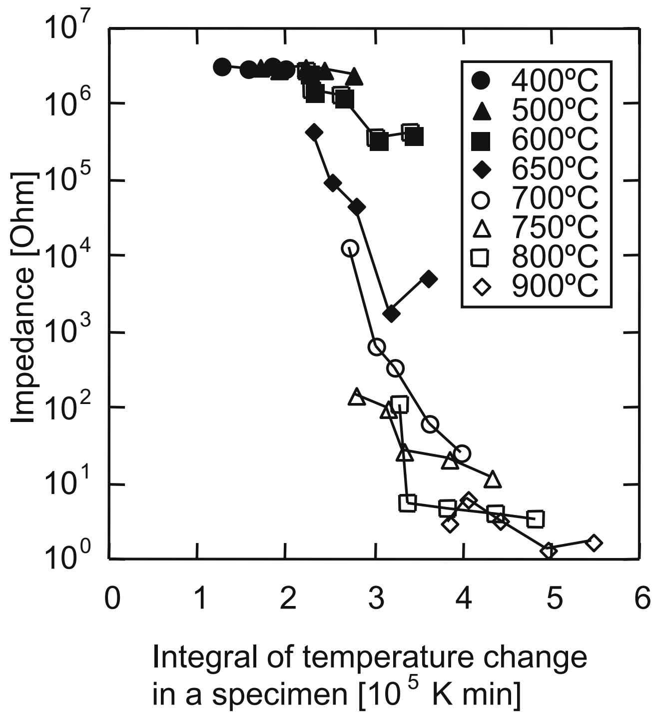

Fig. 6. Relationship between the impedance and the integral of temperature with time in a specimen during heating

over a wide range of frequencies is being developed for our next study.

In the current set of experiments, the yield did not change greatly with the holding time, as mentioned above, and the impedance changed at set temperatures in the range 600°–750°C. Hence, although the yield was easy to measure, the quality of bamboo charcoal could not be estimated from the yield, and electric tests were required.

In making bamboo charcoal, the heating temperature and heating time are the main controlled variables, so we investigated a term composed of both heating temperature and heating time. As the term that influences the impedance, the integral of temperature with time in a specimen during heating, I, is expressed by the following equation:

$$I = \int_0^F (T - \text{r.t.})\text{d}t$$

where T is the sample temperature, r.t. is room temperature, t is the heating time, and F is the time when heating stops. For the sake of simplicity, the specific heat of a specimen is assumed to be constant during heating.

Roughly speaking, when $I \leq 2.5 \times 10^5$ K min, the impedance Z remained high; when 2.5×10^5 K min $\leq I \leq 4.0 \times 10^5$ K min, Z suddenly decreased; and when 4.0×10^5 K min $\leq I$, Z remained low (Fig. 6). However, the Z–I relationship could not be expressed by one curve. This may be because different physical or chemical changes occurred in the specimens. For example, according to Ishihara,[14] when wood is heated in conditions without oxygen, thermal degradation occurs at 60°–200°C, thermal decomposition at 160°–450°C, charcoalization at 260°–800°C, carbonization at 600°–1800°C, and graphitization at 1600°–3000°C. From the above results, the term I may be useful for estimating the approximate value of Z.

Conclusions

The electric properties of bamboo charcoal made at 400°–900°C at hold times in the range 0–3 h were investigated with an alternating current method. The results were as follows:

1. The yield decreased with an increase in the set temperature. The changes in the yield in relation to the holding time were small for most of the set temperatures. The quality of the bamboo charcoal could not be estimated from the yield, and electric tests were needed.
2. The effect of the variation in sample thickness on the impedance could be ignored.
3. The most practical testing method for high accuracy was attaching two plate electrodes to the same surface of a specimen.
4. The impedance was a suitable index for estimating the refining degree of the bamboo charcoal, such as the specific electric resistance.
5. We believe that bamboo suddenly changes from an insulator to a conductor when processed at 600°–750°C for 0–2 h.
6. It is possible that the temperature–time integral term I is useful for estimating an approximate value of impedance Z.

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