Dielectric properties of carbon from cassava starch synthesized from hydrothermal process

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Abstract. Carbon has been synthesized from biomass materials i.e. cassava starch through a hydrothermal process has potential as dielectric material. The carbon synthesized from cassava starch through a hydrothermal process is then made into pellets, then tested capacitance value using a HIOKI 3522-50 LCR HiTESTER using conductive ITO glass as the electrode during measurement. This study aims to determine the dielectric properties of carbon synthesis through measurement on the value of capacitance. Through the measurement of capacitance values are used to determine the value of the dielectric constant. Measurements were performed with the two aspects, namely the measurement of capacitance with temperature and capacitance measurement of the frequency. The test results indicate a change in the capacitance value both to changes in temperature and frequency. While the value of the dielectric constant also shows the dependence on temperature and frequency. Based on the measurement of dielectric constant value of the carbon that was synthesized shows the level of ability of carbon material in terms of storing a charge. The synthesized carbon is a potential material as a dielectric material which can be applied in the manufacture of capacitors.

1. Introduction
Carbon is an abundant element in nature. This element can be found in organic materials such as wood, coal, or natural fibers. Carbon from organic material can be obtained through the process of decomposition of organic compounds called carbonization processes. This process is a process for converting organic material to charcoal by heating in the absence of oxygen, so that the complex compounds that make up the organic material decompose into charcoal with a high carbon content.

One alternative, used in carbon formation is synthesized from hydrothermal carbonization (HTC) process. This has great potential as a promising path to its intrinsic properties, such as high carbon efficiency under low temperature conditions (≤ 200°C) [1]. The HTC process is an environmentally friendly pathway that uses water as a solvent and its products are sealed in a reaction container under a controlled temperature between 100°C and 200°C. The HTC process also has been used for spherical carbon synthesis using sugar or glucose materials as a precursor at low temperatures (≤ 200°C). The HTC process from biomass can provide promising methods for the rational design of a rich family of carbonaceous and hybrid functional carbon materials with important applications [1]. The hydrothermal process is also used for synthesis of multiwall carbon nanotubes at low-temperature [2], and formation of carbon films on carbides under hydrothermal conditions [3]. Carbon also has been applied as a dielectric material, where research on the dielectric properties of carbon has been performed [4,5,6]. Other studies such as dielectric properties of carbon black–epoxy resin composites were studied in the frequency range between 100 Hz and 15 MHz and over a temperature range 23–98°C [7], Dielectric and microwave properties of carbon nanotubes/carbon black filled natural rubber
composites have been investigated in the 1–12 GHz frequency range [8].

In another aspect, carbon can be utilized as test particles in various nanomaterial engineering toxicological studies including nanotubes and fullerenes [9]. Carbon also can be utilized to strengthen the capacitance response in humidity sensors by using Multiwall Carbon Nanotubes (MWCNTs) [10], measuring metal losses based on electrical capacitance [11], and accurate temperature measurements [12], as well as to detect human serum albumin [13]. Therefore, this study focused on studying the dielectric properties of carbon, where carbon is synthesized from biomass materials ie cassava starch synthesized from hydrothermal process.

2. Experimental Method
2.1. Sample Preparation
Four samples were prepared: sample A, sample B, sample C, and sample D. Sample A is 10% by weight of cassava starch without catalyst homogenized using a magnetic stirrer with 450 rpm for 80 minute at room temperature. Sample B is 10% by weight of cassava starch with the addition of 0.8 gram (5 mmol) ferrocene catalyst. Sample C is 10% by weight of cassava starch with the addition of 1.6 gram (10 mmol) ferrocene catalyst. While sample D is 10% by weight of cassava starch material with the addition of ferrocene catalyst as much as 2.4 gram (15 mmol). Samples B, C, and D were homogenized using a magnetic stirrer at 450 rpm for 90 min at room temperature.

Furthermore, each sample is inserted into the autoclave and locked tightly. The temperature and pressure in the autoclave are raised up to 120°C with a pressure of 15 psi for 24 hours. Once the temperature in the autoclave is lowered to room temperature, the sample is removed. The next step, the sample is put into the furnace where the furnace temperature is optimized for several stages. The first stage, the temperature is increased from temperature T$_i$ = 32°C to T$_f$ = 200°C with the temperature rise level of 5.9°C per minute and annealing for 12 hours. The second stage, the temperature increased again from temperature T$_i$ = 32°C to T$_f$ = 300°C with the temperature rise rate 9,22°C per minute and annealing for 12 hours. Once the temperature in the furnace returns to its initial temperature the sample is removed.

2.2. Formation of Carbon Pellets
The carbon formed after the annealing process is made into fine powder. Subsequently, each of the carbon powders was formed by pelleting the carbon powder into the cradle and pressed by pressing at a pressure of 20 MPa for 30 minutes.

2.3. Capacitance Measurement
Measurement of capacitance value in each sample was done using HIOKI 3522-50 LCR HiTESTER tool which used conductive Indium tin oxide glass (ITO) as the electrode during measurement. Measurement of capacitance is intended to know the value of capacitance and to calculate the dielectric constant value of carbon synthesis both to temperature and frequency.

3. Results and Discussion
Dielectric material is a material which has no free charge which has an important effect on the electrical properties of the material. Dielectric material is very important in electricity because some properties of dielectric materials that can store electrical charge, pass the current back and forth and hold direct current. The dielectric constant is the ratio of the capacitance value of the capacitor in the dielectric material to the value of the capacitance in a vacuum. The dielectric constant is obtained by the equation of capacitance $C = \frac{\varepsilon \varepsilon_0 A}{d}$, where $\varepsilon$ is the dielectric constant of the material between two sheets of conductors having area A and separated by distance d [14].

Capacitance is a measure of "capacity" of charge storage for certain potential differences. In this research, the measurement of capacitance at a certain voltage is 1 Volt, because the potential difference is proportional to the charge so that the capacitance is independent of charge or voltage. Identify the dielectric properties of the carbon by measuring the carbon capacitance of both the frequency change
and the given temperature change. The measurement of capacitance to frequency is shown in Figure 1.

![Graph showing capacitance to frequency change](image1)

**Figure 1.** The characteristics of capacitance value to frequency change.
- Sample A (carbon without catalyst),
- Sample B (carbon with a catalyst of 0.8 gram),
- Sample C (carbon with a catalyst of 1.6 gram),
- Sample D (carbon with a catalyst of 2.4 gram).

Each sample shows the frequency dependence of the capacitance (figure 1). Capacitance increases at a lower frequency whereas at higher frequencies the capacitance tends to decrease and is almost constant in each sample. This is due to the packaging density in each sample [15]. Capacitance also shows a dependence on temperature which is characterized by an increase in capacitance value at higher temperatures, as shown in figure 2.

![Graph showing capacitance to temperature change](image2)

**Figure 2.** The characteristics of capacitance value to temperature change.
- Sample A (carbon without catalyst),
- Sample B (carbon with a catalyst of 0.8 gram),
- Sample C (carbon with a catalyst of 1.6 gram),
- Sample D (carbon with a catalyst of 2.4 gram).

Increased capacitance occurs due to the polarization of the dielectric material i.e., the movement of electrons, ions, and polar molecules within the dielectric caused by the presence of an electric field. Increased capacitance at high temperatures can be due to space charge polarization and orientation polarization. The polarization of the space charge occurs due to the separation of space charges which are free charges in the dielectric space. With this process there is a similar charge collection on both sides of the dielectric. While orientation polarization occurs in the material by forming a permanent dipole moment. These permanent dipole will tend to orient themselves parallel to the electric field, but not all dipoles will be parallel to the direction of the field.

Increased capacitance to frequency can also be caused by electronic polarization and ion polarization on dielectric materials. Electronic polarization occurs due to electron cloud shifts in atoms or molecules due to an electric field where the center of positive and negative electric charges that initially coincide into a separate dipole to form. This separation of the central point of charge continues...
until there is a balance with the electric field causing it. Dipole is formed is not permanent dipole, meaning that dipole formed during electric field effect only. If the electric field is lost then the center points of the load again coincide again. If the given field is a unidirectional field, the dipole is formed almost instantaneously with the presence of an electric field. While the ion polarization occurs due to shift of ions opposite the sign by the electric field influence. As with electronic polarization, the dipole formed in ionic polarization is also a non-permanent dipole. However, ion polarization occurs more slowly than electronic polarization. When a unidirectional field is taken, it may take longer to reach a balanced state. Capacitance has a strong dependence on temperature, whereas capacitance tends to a constant value at higher frequencies. This anomaly is thought to originate from oxygen shrinkage out of the sample during the heating process [15].

Characterization of the dielectric constant to frequency is shown in Figure 3.

![Figure 3](image)

**Figure 3.** The Characteristic value of dielectric constant to frequency change.

Sample A (carbon without catalyst), Sample B (carbon with a catalyst of 0.8 gram), Sample C (carbon with a catalyst of 1.6 gram), Sample D (carbon with a catalyst of 2.4 gram).

Based on figure 3 above shows that the dielectric constant decreases with increasing frequency. The pattern is similar to the characteristic capacitance to the frequency. This occurs because between the capacitance and the dielectric constant have a linear relationship. So that the increase of dielectric constant factor is influenced by the increase of capacitance value owned by carbon of synthesis. While the characterization of dielectric constant to temperature is shown in Figure 4.

![Figure 4](image)

**Figure 4.** The Characteristic value of dielectric constant to temperature change.

Sample A (carbon without catalyst), Sample B (carbon with a catalyst of 0.8 gram), Sample C (carbon with a catalyst of 1.6 gram), Sample D (carbon with a catalyst of 2.4 gram).
The characteristics of the sample dielectric constant also show an increase in temperature. All samples show the same pattern to temperature. Since the dielectric constant has a linear relationship with the capacitance, the characteristic of the dielectric constant to temperature also shows the same pattern with the characteristic pattern of capacitance to temperature. The high dielectric constant possessed by the sample shows the higher capability possessed by the sample in terms of storage of charge.

Based on the change of capacitance value and dielectric constant to temperature and frequency, the carbon which is synthesized from biomass material is dielectric material which has dependence both on temperature and frequency. This will certainly be very useful in the development of capacitors of carbon-based dielectric materials. On the other hand, the use of the capacitance of the dielectric material essentially also has been applied to the sensor field. Some aspects include humidity sensor [11], and for detecting human serum albumin [13].

4. Conclusion
The carbon synthesized from the biomass material through the hydrothermal process is essentially a dielectric material which still has a dependence on the temperature and the given frequency. The synthesized carbon is a potential material as a dielectric material which can be applied in the manufacture of capacitors. This can be seen from the characteristic change of capacitance value and dielectric constant both to the treatment of temperature change and frequency change. The value of the dielectric constant also indicates the ability of the carbon material to be synthesized through the hydrothermal process in terms of the charge storing capacity.

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