



# The Effect of Film Thickness and Sintering Temperature on the Electrical Performance of Carbon Electrode

Ozuomba J. O.<sup>1</sup>, Okoli L. U.<sup>2</sup>, Ekpunobi A. J.<sup>3</sup>

<sup>1</sup>Department of Physics and Industrial Physics, Madonna University, Elele, Nigeria.

<sup>2</sup>Department of Physics, Federal College of Education, Technical, Umuoze, Nigeria.

<sup>3</sup>Department of Physics and Industrial Physics, Nnamdi Azikiwe University, Awka, Nigeria

## ABSTRACT

Four samples of carbon electrode were fabricated by sol-gel process. Two of the samples were fabricated from a carbon paste fabricated from powdered activated carbon (PAC) while the other two were produced from an equal mixture of powdered activated carbon and natural graphite powder (NGP). Electrode deposition was achieved through doctor blading and we were able to vary film thickness through single and multiple layer deposition. Our investigation revealed that the electrodes produced from a mixture of PAC and NGP have low sheet resistance when compared to those fabricated from PAC only, while thicker films have lower sheet resistance. Also, sheet resistance of carbon electrode increases with sintering temperature and a very significant increase was observed after sintering at 450°C.

**Keywords:** *Sintering, carbon, electrical properties, sol-gel.*

## I. INTRODUCTION

Electrical properties of sintered carbon electrode depend on sintering temperatures [1,2]. Sintering involves heating a material in a sintering furnace below its melting point (solid state sintering) until its particles adhere to each other [3]. Sintering is necessary to reduce porosity and enhance properties such as strength, translucency and thermal conductivity.

Various carbon-based materials (carbon black, carbon nanostructures, glassy carbon, carbon films, activated carbon) are used as electrodes for supercapacitors [4]. Such interest on carbon-based materials is related to their high total surface area, porosity, high temperature stability, conductivity and pore size of carbon material [4-8]. Carbonaceous materials has played a major role in the industrial electrochemical production of many chemical materials, such as: chlorine and caustic, aluminium metal, and organic chemicals [8]. Carbon is formed into solid structures that serve as the electrode sites where electrochemical reactions occur and products are formed. The key ingredient in fuel cells is the use of electrode materials that facilitate (catalyze) electrochemical reactions. Two major types of fuel cells rely heavily on carbon, usually in the type of graphitized carbon [9]. These fuel cells are referred to as the phosphoric-acid fuel cell (PAFC) and the polymer-electrolyte-membrane fuel cell (PEMFC). Also, carbon electrodes are of great importance for dry batteries and dye sensitized solar cells [1,5,10-12].

Carbon has been used as an electrode material due to its wide window of electro-chemical stability and its ability to combine with itself to form varying structural forms (allotropes) [13]. This ability (called catenation) allows us to develop novel fabrication routes to create carbon platforms starting from most organic

precursor or polymers on hand. The sol-gel process provides a versatile means for the production of inorganic and inorganic-organic hybrid materials via the hydrolysis and condensation of suitable metal alkoxides [14]. The sol-gel process is a wet-chemical technique (also known as chemical solution deposition) widely used recently in the fields of materials science and ceramic engineering [15]. This method has gained interest, as it presents many advantages: the use of very simple equipment and a lower capital investment requirement, the high homogeneity of the final thin films, the possibility of using a range of different substrates and the control of the microstructure and density of the thin films [14,16-18]. The sol gel processes are particularly efficient in producing thin, transparent, multi-component oxide layers of many compositions on various substrates, including glass [1,2,3-5,14].

Powdered activated carbon (PAC) and a kind of natural graphite powder (NGP) were used to obtain our gel-like diphasic system (carbon paste) while electrode deposition was enabled through doctor blading. Four sample electrodes of varying composition and thickness were fabricated. Each electrode was sintered at different temperatures and the sheet resistance was measured after each sintering. This project is necessary due to the numerous applications of carbon electrode.

## 2. EXPERIMENTAL

### 2.1 Reagents

Tin (ii) chloride, carboxy ethyl cellulose (NATROSOL), 25% hydrochloric acid, powdered activated carbon (PAC) and a kind of natural graphite powder (NGP). High purity de-ionized water was used to prepare the solutions at a temperature of 25±2°C. The



chemicals were of analytical grade and used without further purification.

## 2.2 Apparatus and Physical Measurements

Ainsworth DE – 100, Max 100g, e = 0.0001g chemical balance was used to weigh the chemicals. Hot air blower was used to dry the carbon paste electrode immediately after deposition before thermal annealing using electric hot plate. Carbolite 201 tubular furnace was used for the sintering process. Measurement of sheet resistance was done using dual-Pro 301 auto calculating four-point probe resistivity test system.

## 2.3 Electrode Fabrication

We dissolved 12g of tin (ii) chloride in 50ml de-ionized water and titrated with about 1ml of 25% HCl. The solution became colourless and there was a noticeable fall in temperature. Then 2g of NATROSOL was dissolved in 50ml de-ionized water and stirred properly. We mixed the two solutions and stirring continued. This mixture was heated to 300°C using a hot-plate. Stirring continued as the heating lasted for about 39 seconds. The resulting solution (about 16ml) was allowed to stay overnight at room temperature. It was further heated on the following day for about 7 minutes to get the polymeric colloidal sol of about 12ml.

The first carbon paste (Sample A) and Sample B were fabricated by mixing well ground PAC and the colloidal solution in the ratio of 2.0g/ml and 3.0g/ml respectively. Also, equal mixture of well ground PAC and

NGP was used to obtain another carbon paste in the ratio of 3.0g/ml which was used to obtain Sample C and Sample D. Electrode was deposited onto FTO glass substrate through doctor blading. Samples A and C were fabricated using single layer deposition while Sample B and Sample D were obtained by double layer deposition [1,19].

## 2.4 Thermal Treatment

A hot air blower was used to dry each carbon paste electrode for about three minutes immediately after deposition. Using an electric hot plate, the carbon electrode was subjected to thermal annealing at 150°C for five minutes. Immediately after annealing, the electrode was inserted into carbolite 201 tubular furnace for the sintering process. Each electrode was sintered for 15 minutes at different temperatures ranging from 200°C to 450°C. The sheet resistance was measured after each sintering process using dual-Pro 301 auto calculating four-point probe resistivity test system.

## 3. RESULTS AND DISCUSSION

Figure 1 is the effect of sintering temperature on the sheet resistance of Sample A. The sheet resistance increased gradually from 77ohms/square to 138ohms/square between 200°C and 400°C but increased to a high margin of 259ohms/square after sintering at 450°C.

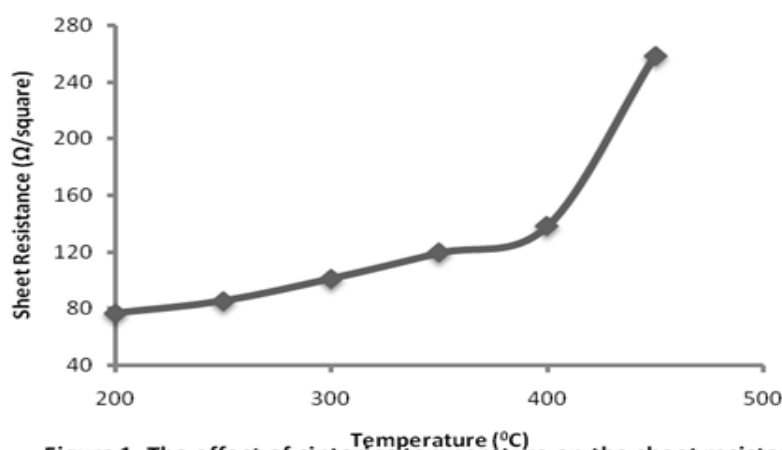
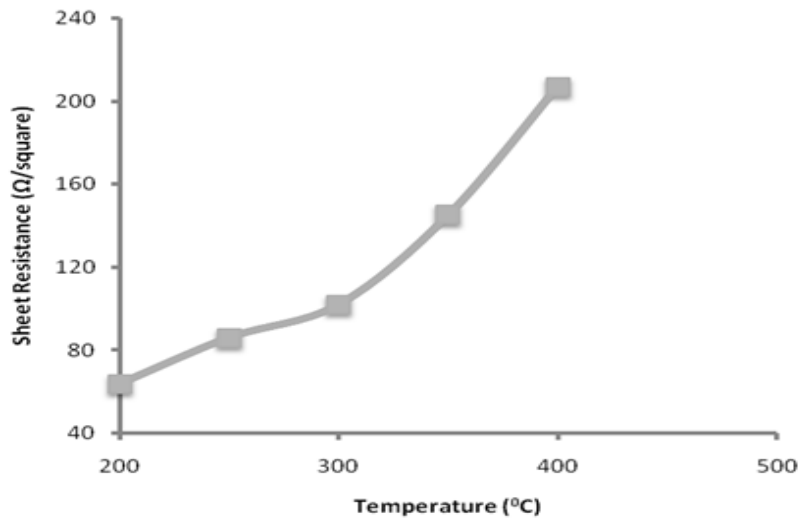


Figure 1. The effect of sintering temperature on the sheet resistance of Sample A

Figure 2 shows the effect of sintering temperature on the sheet resistance of Sample B. The sheet resistance increased almost linearly from 64ohms/square at 200°C

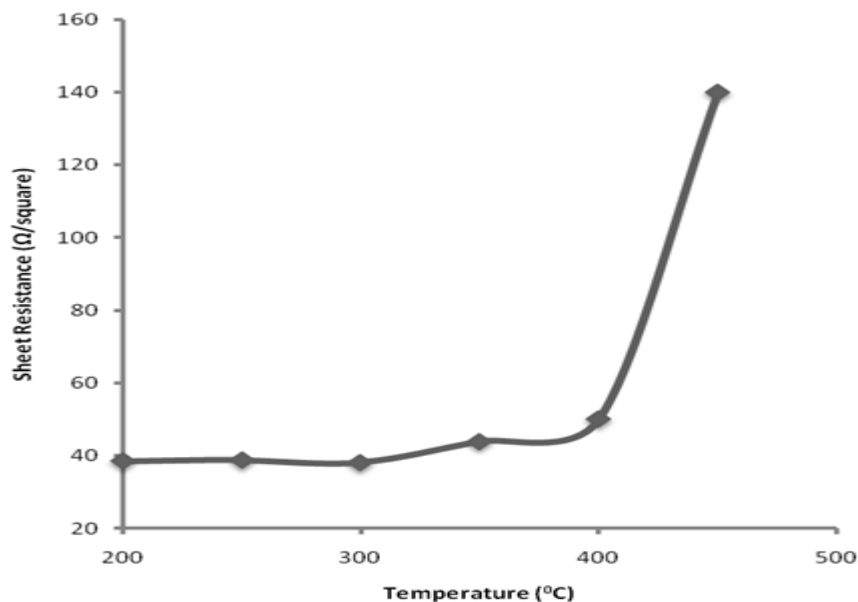
to 58800C at 4500C. The sheet resistance after sintering at 4500C was comparably very high.



**Figure 2. The effect of sintering temperature on the sheet resistance of Sample B.**

The dependence of sheet resistance on sintering temperature for Sample C is shown in Figure 3. The sheet resistance was almost steady from 200°C to 350°C but a sharp increase was also recorded after sintering at 450°C. The effect of sintering temperature on the sheet resistance of Sample D is shown in Figure 4. The

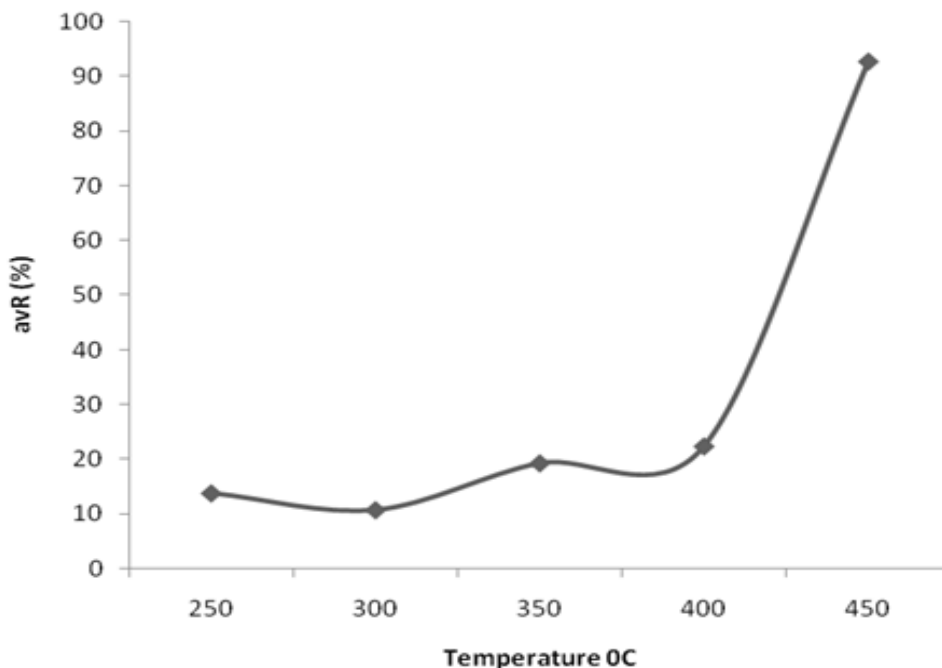
electrical performance of Sample D was generally stable between 200°C and 300°C. The sheet resistance increased gradually from 38 ohms/square to 50 ohms/square between 300°C and 400°C but sky-rocketed to 140 ohms per square after sintering at 450°C.



**Figure 4. The effect of sintering temperature on the sheet resistance of Sample D.**

The average effect of sintering temperature on the sheet resistance of the four samples is shown in Figure 5.

The percentage change in the average sheet resistance varied from about 14% at 250°C to 22% at 400°C but sky-rocketed to 93% at 450°C.



**Figure . Percentage of average change in sheet resistance (avR) versus sintering temperature**

#### 4. CONCLUSION

The experimental results show that the sheet resistance of carbon paste electrode depends on composition, film thickness and sintering temperature. The sheet resistance reduced significantly when graphite was added to the activated carbon and further reduction can be achieved through multiple layer deposition. Also, the electrode formed from a mixture of powdered activated carbon and natural graphite powder showed more stability under sintering at different temperatures. Generally, sheet resistance of the carbon electrode increases with increase in sintering temperature. 400°C was found to be the critical sintering temperature for the sol-gel derived carbon paste electrode.

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