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Fabrication, Electrical characterisation and modelling of iron –clay composite resistor

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ABSTRACT

Iron-clay based composite resistor of varying iron content and aspect ratio have been fabricated using a pressure of about ($2.04x10^8$) Nm^{-2} for all the resistor moulded, expect in the case where the pressure was varied with four different pressures of about $1.28x10^8Nm^{-2}$, $0.66x10^8Nm^{-2}$, $2.04x10^8$ NM^{-2} and $2.41x10^8Nm^{-2}$ with composition of 70% iron, length of 15mm and particle size of 0-25µm. The effect of clay particle size was also investigated using particle size ranging from 0-250µm, 250-400µm, 400-500µm and 500-850µm. Iron powder was used as the conductive element while the clay powder served as insulating and binding element. The fabricated resistor all have an average diameter of 3mm; for those moulded with particle size of 0-25irm, the length are varied between 5mm and 25mm increasing in 5mm and composition of 95%, 90%, 85%, 80% and 75% iron content. The resistors were investigated for influence of composition, firing time, firing temperature, length particle size and moulding pressure on their electrical properties. The results show that the electrical resistance increases with increasing resistors length and increasing the diameter of the clay particle size. However, the electrical resistance falls rapidly with increasing firing temperature, firing time, moulding pressure and also with increasing functional composition (iron composition). The modeled results also shows that polynomial of the 2[nd] order best describes the results obtained.

Introduction

Composite resistors or cermets consist of a thorough mixture of a conducting phase and insulating phase. The electrical properties of exhibited by composites, which are unimaginable in any of the constituent phases makes them extremely valuable for use in many electronics circuits Ayodele and Akomolafe (2005). Electronic conduction is a phenomenon in material that is brought about by the response of electrons in a given bands to external field, magnetic field and temperature gradient . it is regulated by collision with impurities and other crystal imperfection. Some work had tend to explain in qualitative and quantitative terms the electrical properties of the cermets resistors made from combination of clay and metal powder these include: Prudenziati and Acquab (1994), Morten et al, (1994), Akomolafe and Oladipo (1996), Arfonte et al (1997), Ayodele and Akomolafe (2005), Babalola (2010). The electronic conduction includes several possibilities which include; tunnelling of electrons between near neighbouring grains assisted by resonant centres in the intergranular material, hoping of electrons from to conductive grains and localized states in the glassy matrix, Babalola (2010). The works of Akomolafe and oladipo, (1996), Ayodele and Akomolafe (2005), Babalola (2010) have shown that this type of resistor exhibited a negative TCR, hence this work made an attempt at explaining some properties of the this type of composite resistor and defining a model that best suites the type of curves in terms of regression analysis.

Materials and methods

Materials:

The iron clay-based composite resistor has the following characteristics: The conductive element which is iron powder manufactured by M & B Chemical laboratory England, Clay obtained from a River-Bed at the flower garden in Ilorin Nigeria, was used as for insulation and binding. The clay was carefully selected for its homogenous physical properties. Graphite electrode grounded flat at the ends were used to form the terminals for the moulded resistor since it was impossible for solder to stick to the terminals of the moulded resistor. This was so because the flux in the soldering lead was not compatible with the iron-clay composition.

Fabrication and test equipment

A moulding press which can exert a high compression force was used. The press was calibrated using a weighing scale capable of measuring a maximum force of 1200N. A jack was used to compress the spring and the compression was taken for every 150N interval. The force constant of the spring was determined to be $2.67x10^4Nm^{-1}$ approximately.

The resistors were fired in an electric oven at various temperature up to the maximum of 1000^oC. Resistance at various annealing temperature were measured using a multimeter that consist of both digital and analogue scales. An electronic digital weighing digital weighing balance and a sieve were used to weigh and sieve the clay to the desired particle size respectively.

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Resistor production

There are three major steps in the production of this type of resistor as explained below.

1.Material selection and processing.

The selected clay was washed in a bucket of water so that it formed a suspension. The water clay mixture was sieved to remove stone and debris. The water clay suspension was left for a week so that the clay will form a sediment inside the water. The top water was then drained off and the clay sediment was dried in the sun for two weeks. The dried clay was grounded on a clean cemented surface using a clean hard stone. The powder produced was sieved to the desired size.

2. Mixing stage

The iron and clay powders were mixed in five different proportion of clay to iron proportions and percentage clay/iron ratios of 5/95, 10/90, 15/85, 20/80 and 25/75 were obtained. Twenty grams of the mixture of the iron and clay were measured using the digital weighing balance with accuracy of ±0.001g.

3. Moulding stage

To reduce the effect of too much water content on copper-clay resistor, the content of the water was made low. The iron clay mixture was sprayed with just enough water to cause compaction and fusing together under high pressure .The mould was greased slightly to facilitate easy removal. After greasing the eight holes in the mould were filled with semi-dry-iron-clay mixture and then put under the press at moulding pressure of $2.04X10^{8}Nm^{-1}$. The resistor produced had an average length of 30-35mm and a constant diameter of 3mm and were then cut to different length of 5mm,10mm, 15mm, 20mm and 25mm.

Furnacing

Five resistor each were chosen for different length and composition . their resistance was measured at room temperature with the multimeter and graphite electrode. They were then put in the furnace, set a 100oc and fired at the peak temperature for three hours. The resistor was then furnace cooled to room temperature and the resistance values were then recorded again. All readings below below 300^{o}c were discarded because the resistance values for resistors especially those with large clay content and length >5mm could not be read on the meter. The average resistance value for the five resistors were taken and the resulting graphs were shown in th result

Results and discussion

Variation of resistance with firing time

In resistor a little alteration in a peak firing temperature or a change of three or four minutes in the peak zone can cause a remarkable change in the final resistance. To this the firing temperature and time are chosen to ensure that correct amount of reactions occurs in the resistor. Figure 1, shows the variation of resistance with firing time at firing temperature of T_f 500^{o}c and 600^{o}c for resistor with 75weight percent of Fe and length 15mm. It could be seen that the values of resistance fall with increasing firing time, this fall in resistance is rapid during the early part of the firing between 30minutes and 90minutes, and proceeds afterward at slower rate. This observation may be due to the fact that a large amount of reaction such as removal of organic and structural defects, and formation of required chemical composition towards the final electrical resistance might have occurred at the early stage of the firing profile. The completion of this reaction occur later during the firing process. This result in the reduced rate of fall of resistance with firing time which levels off at firing time ≥120minutes as observed.

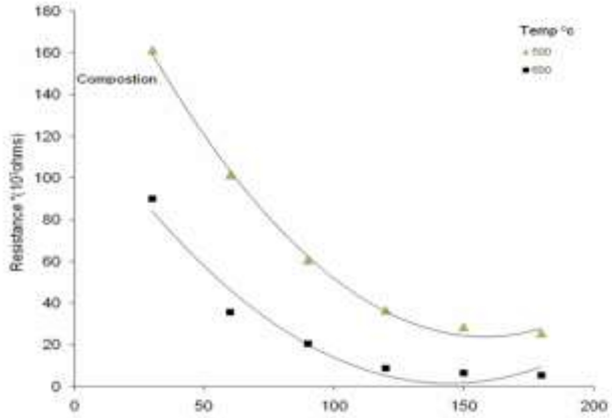

Fig. 1. Variation of resistance with firing time with resistor length of 15mm and 75%Fe at various firing temprature in °c

Variation of resistance with composition

In any composition resistor the composition of the functional material (conductive phase or metal) to the insulative phase(clay) has a great effect on the final electrical properties of the cermets resistors. Figure 2a and 2b shows the variation of resistance with composition for constant length and constant temperature respectively. From the results it shows that resistors have high resistance at 75% composition and lowest at 95% composition of iron. This can be attributed to the fact that the resistors with 75% iron composition contains the highest percentage of clay particles and otherwise for 95%Fe. With this high content, the structure with 75% Fe is now that of more of insulative particle dispersed in a conducting medium. Since the electrical conductivity of a resistor is a property which depends on upon electron phenomenon. The more freely an electron can move in a material, the lager the conductivity value of that material.

However, in a bulk resistor, the resistance is due to the fact that collision of conducting electron with vibrating lattice atoms, impurities and defects. The modifications to the bulk conductivity of the resistor due to these scattering centres depend on their concentration. This is because a high concentration of scattering centres will tend to reduce the free path of the electron and increase the magnitude of the electron momentum loss in the field direction after collision with centre. This effect is more pronounced in composition with high insulating content. From the pattern of the non linear variation of resistance with composition, the relevant roles played by fabrication parameters in determining the final electrical resistance of the resistor are evident. The values of the resistance are larger for resistors fired at lower temperature and having longer length.

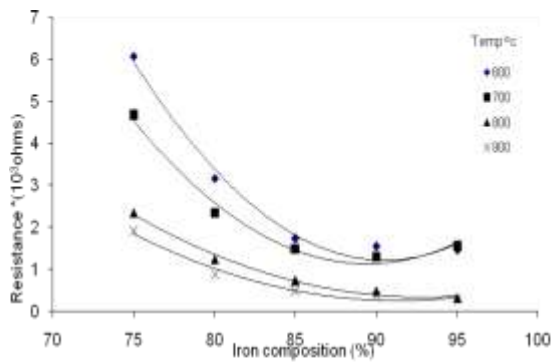

Fig. 2a. Variation of resistance with iron composition at various firing temprature in °c and resistor length of 15mm

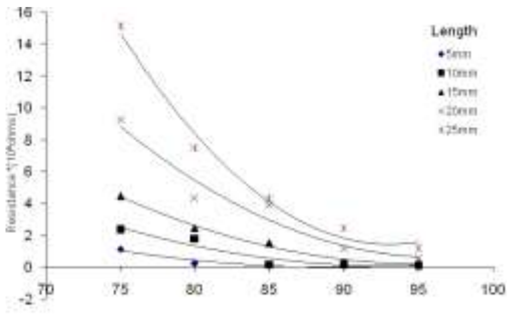

Fig. 2b.. Variation of resistance with iron composition for various resistors length at firing temprature of 600°c

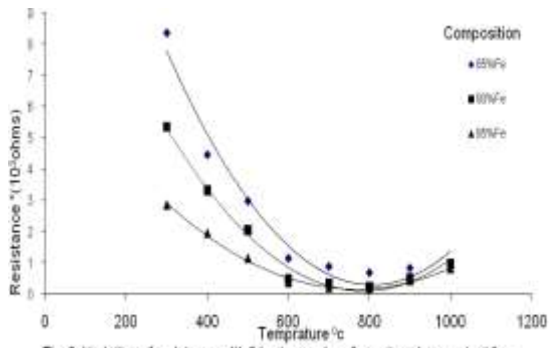

Fig. 3. Variation of resistance with firing temprature for various iron content for resistor length of 10mm

Variation of resistance with firing temperature

As earlier stated, it is known that annealing seriously affects the electrical properties of composite or cermets resistors. A number of structural defects such as impurity atoms, vacancies and interstitial, dislocations and grain boundaries are produced, the aggregate of these imperfections makes the resistivity of the resistors higher than their bulk counterpart (Akomolafe and Oladipo 1996). Thus, we have decided to investigate the variation of resistance with the peak firing temperature T_f for Fe-clay composition resistors. Since it is known that the annealing time and firing peak temperature help in the formation of the correct chemical structure in the correct proportions for the required resistance and help in burning out any resin type materials in the organic vehicle left in the resistor, and also provides cohesion in the in the bulk resistance itself. Figure 3 shows the effect of firing temperature against resistance for resistor length of 10mm. It can be seen that for all concentration, figure 3 have its minimum resistance value at 700^oc and 800^oc. The comparatively high resistance observed at $T_f < 400^oc$ could be attributed to the incomplete sintering of the iron-clay based composite mixture as a result of low T_f. It should be noted that the possibility that the sintering might have occurred at different temperatures explains the reason why the resistance were observed to fall at different rates as seen in figure3. However ,the sudden increase in the resistance observed for $Tf>800^oc$ is attributed to the furnace atmosphere oxidation which causes the increase in the resistance value of the material. These oxides formed with the iron could be Fe_2O_3, Fe_3O_2 or FeO (Akomolafe and Oladipo, 1996). It can also be observed that the resistance value of the material is falling with increasing temperature between T_f equal 300^oc and T_f 700^oc and this could be attributed to the burning out of the liquid vehicles and the sintering of individual particles to form a continuous metal conduction path through the cermets structure. According to according to (Babalola, 2010) quoting the Ziman(1960) who observed that using the free electron theory, the resistivity may be obtained in terms of the carrier mean free path such that

$$\rho = \frac{\left(\frac{3}{8}\right)^{3} h}{q^2 n^{2/3} \lambda} \text{, --} \quad (1)$$

where, h is the plank constant, λ is the carrier mean path, and q is the charge of the electron. He concluded that the increase in the resistance of the composite resistors with increasing annealing temperatures at annealing temperature exceeding 700^oC is explained, since it is expected that narrowing the conduction path will reduce λ and thus increase the resistivity ρ.

Variation of resistance with length

For a cermets resistor, it is expected that as the length of the increases, the resistance of the resistor should also show an increase due to the fact that impurity, imperfection etc should increase with length of the resistor there by increasing its resistance. The variation of resistor length on the normalized resistance R_s is known as size effect. In this case is given by Akomolafe and Oladipo(1996)

$$R_s = \frac{R_n}{R_{n(25mm)}} \quad (2)$$

where

R_n = Resistance of a given length of a resistor

$R_{n(25mm)}$ = Resistance of the maximum length of cermets resistor

The effect of the resistance length on the normalized sheet resistance is shown in fig. 4a and 4b for various iron concentration at firing temperature of 600^oc and for various temperature at 75% iron concentration respectively. It was observed that the work is in the direct agreement with Babalola (2010) who observed that direct size effect (namely, lower effective sheet resistance for short resistance) is exhibited for composite resistor at various annealing temperature irrespective of the annealing temperature, it is was also observed that the sheet resistance is smaller in resistor with large Fe composition irrespective of the annealing temperature.

A material exhibiting size effect will either present an indirect or direct size effect but not both. For the resistors studied it was observed that they exhibit direct size effect since the sheet resistance for the shorter length are lower than those of the longer length. This had been reported by prudenziati et al (1991), Morten et al (1991), Hrovat et al (1986),

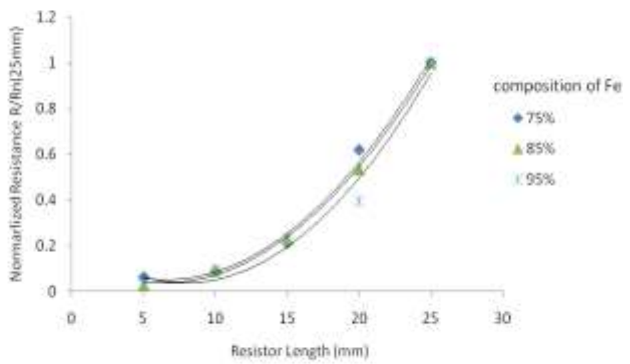

Figure 4a:Variation of Normalized resistance with length for various iron compostion at firing temperature of 600°c

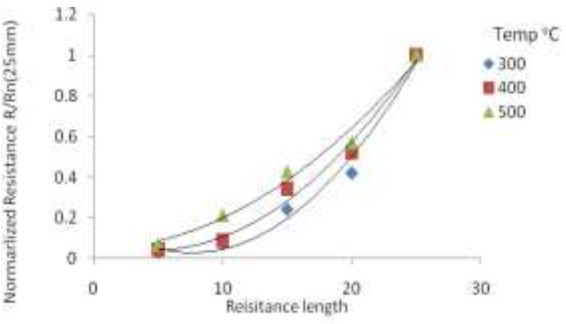

Figure 4b: Variation of Normalized resistance with length for various temperature for 75% iron concentration.

The resistor studied are found to increase non linearly with length. The non-linear mode of variation of resistance can be attributed to the mode of control of the amount and distribution of impurities and defects still present in the resistor for each length after firing. fig. 5a shows the Variation of resistance with length for various iron composition at firing temprature of 800^{o}c and fig. 5b shows the variation of resistance with length for various firing temprature at 95% Fe. It was found that the resistance of the material increase non-linearly with the

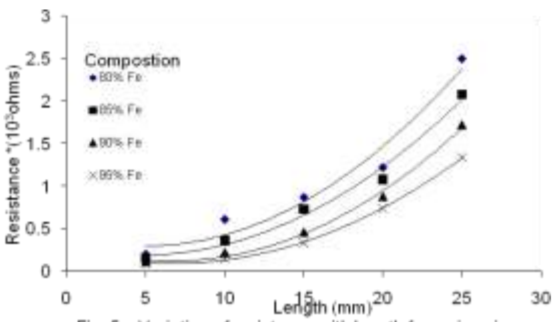

Fig. 5a. Variation of resistance with length for various iron composition at firing temprature of 800°c

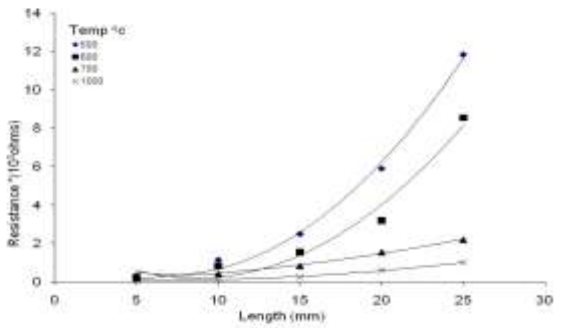

Fig. 5b. Variation of resistance with length for various firing temprature at 95% Fe

resistor length .The increasing resistance with increase in resistors length might find an explanation in the electrical conduction mechanism in thick-film resistors described by electron percolation theory. The structure consists of conductive grains make interaction through electronic layers so thin that electron is able to tunnel through the layer. However, some inhomogenuity may be introduced into the structure as the length of the resistor is increased. This may result in larger separation between conductive grains thus reducing the possibility of electron tunneling and percolation. The resistance will then increase. There are numbers of reason why resistor may not give the resistance- length variation predicate from theory. These are as given by (Akomolafe and Oladipo, 1996) include:

(a) A real modification of the microstructure and composition of the resistor due to some chemical reactions between the resistive material itself, and with the metallic termination on the other hand.

(b) Formation of structural defects such as micro cracks and variation of resistor thickness with length may result due to difference in the coefficient of constituent material or due to

(c) Formation of a partially insulating barrier layers may occur as a thin oxide film when the conductor material oxidizes. The effect of this is force all the currents to pass through a few higher conductivity network covering a small portion of the resistor resulting in high local current density.

Variation of resistance with particle size

It is expected that the particle size of the insulation phase would affect significantly the compactness of the cermet material, transmission of pressure, vacancies, grain boundaries etc. These would in turn affect the final electrical properties of the cermet material. The fraction/percentage composition of the insulating phase to conductive phase determines the bulk dc resistance and electrical properties of metal-clay based cermets(Babalola 2008). Three major parameters that had been identified to affect the bulk resistance of cermets are: constriction of resistance associated with the constriction of the electron flow through the small area where any two conducting particles meet (Ruschau et al, 1992), tunneling resistance associated with the insulating layer coating the conducting particles and the intrinsic grain resistance of each particle, the constriction of resistance and the tunneling resistance of particle-particle contacts have also been known to depend on the conducting particles size(Ruschau, 1972, Holm, 1967, Ayodele and Akomolafe, 2005, Babalola, 2008). The resistance/resistivity of the iron-clay cermet resistor is observed to increase non-linearly with the clay particle size for all the firing temperatures considered; this variation is show in figure 6a and 6b which shows the variation of the resistor resistance with average clay particle size for various firing temperature. The increase in resistivity observed in this work may be attributed to the following reason.

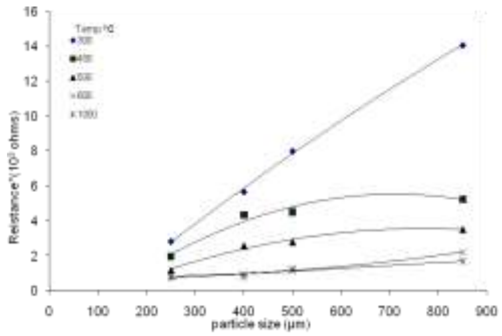

Fig. 6a: Variation of resistance with paricle sizes. With 95% Fe and resistor length of 10mm at various firing temprature.

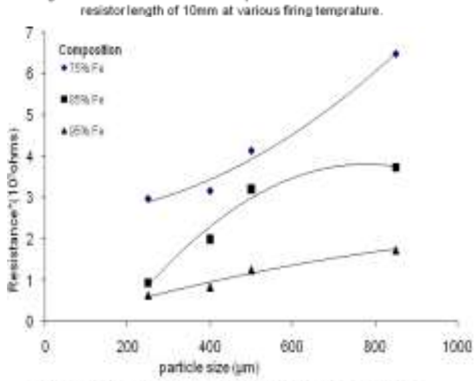

Fig.6b Variation of Resistance against particle size for various iron composition with a resistor length of 10mm at 1000°c firing temprature

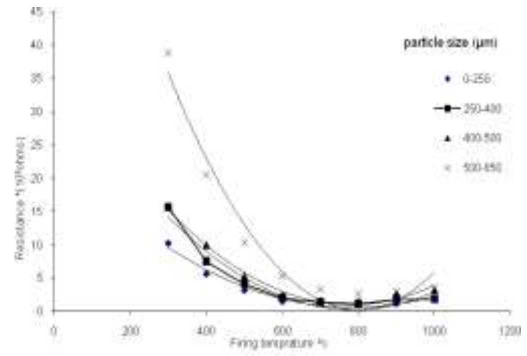

Fig 6c. Variation of resistance with temprature for different particles sizes with resistor length of 10mm and 85% Fe

(1) Larger clay particle size would reduce the compacting pressure generated in the resistor thereby increase the final resistance of cermet resistor (Akomolafe and Oladipo,1996 and Babalola, 2008)

(2) Larger clay particle size would result in more structural defect such as vacancies and fracture since the bond between the binder and the conductive element would not be as strong as when the binder has a smaller particle size.

(3) Larger clay particle size may increase the porosity of the cermet material thereby enhancing the easy diffusion of gases specifically oxygen into the cermet structure hence the increase in oxidation of the metallic conductive phase is promoted. This will lead to the formation of higher valued resistors. Fig. 6c also confirms this result. It shows the variation of resistance with firing temperature T_f for resistor of different clay particle sizes. It can be observed that the average resistance / resistivity increase with increasing particles size of all the firing temperature. For all the particle sizes it could be observed that the effect is more pronounced in higher clay particle size and less in lower clay particle size. This may be attributed to the fact that the smaller clay particle size has the effect of shielding the iron-powder from oxidation in the furnance

Variation of resistance with pressure

Resistors are composite conductive materials whose electrical resistance is also related to their moulding pressure. Fig. 7a and. 7b show the variation of resistance with pressure for various temperatures. It is shown that for all the temperatures, the resistance decreases with increasing moulding pressure. The electrical conductivity of a resistor is a property which depends upon transport phenomena(Akomolafe and Oladipo, 1996). The more freely an electron can move in a material, the larger the conductivity value of the material.

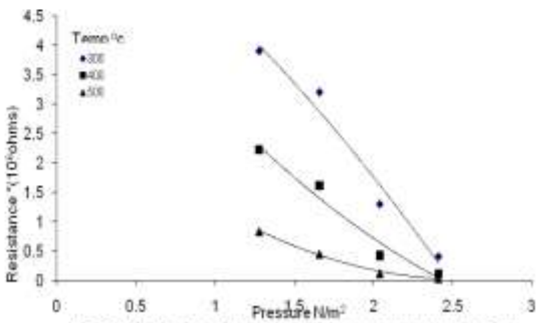

Fig 7a. Variation of resistance with moulding pressure at various firing temprature with a resistor length of 15mm and 75% Fe

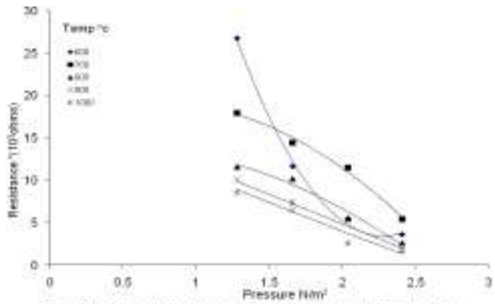

Fig. 7b. Variation of resistance with moulding pressure for various firing temprature with resistors length of 15mm and 75% Fe

The low resistance observed under the high pressure is due to the fact that there will be better interaction between the metal grains and reduction in the porosity of the cermet materials thereby reducing the diffusion of gases which may cause increase in the resistance value. As a result of metal grains contact, the effect of percolation and tunneling will be more pronounced and hence this leads to increase conductivity pressure and low conductivity with low compacting pressure.

Modelling

The composite resistors studied in this work have been modelled using the simple regression analysis method. The obtained model is well represented by polynomial degree of the 2nd order noting that the model is well explained between 91 and 99.9% level. In comparing with other functions such as: exponential, power, logarithmic, linear it is observed that the polynomial function give the best R^2 value for 92% of times and since this difference is always negligible at about 0.02 or less, the polynomial function was used to describe all the results. The results are presented in the following tables for all the figures respectively. The general polynomial equation is given by

$$R(Y) = AX^m + BX^n + C \qquad 3$$

where; A,B,C are constant given in the tables, m = 2, and n =1, R(Y) is the value of resistance for a given function and X is the variable representing variable on the Abascia axis.

Table 1. Table of the modelled equation of the variation of resistance with firing time with resistor length of 15mm and 75%Fe at 500 and 600°C

Temp(°c)	A	B	C	R^2
500	0.0083	-2.6141	230.05	0.9977
600	0.0063	-1.8243	133.05	0.9618

Table 2a. Table of the modelled equation of the variation of resistance with iron composition at various firing temprature in °c and resistor length of 15mm

Temp °c	A	B	C	R^2
600	0.0197	-3.5581	162.22	0.9871
700	0.0164	-2.9383	132.5	0.9863
800	0.0061	-1.1405	53.278	0.9894
900	0.0059	-1.0885	49.82	0.9775

Table 2b. Table of the modelled equation of the variation of resistance with iron composition for various resistors length at firing temprature of 500°c

Length (mm)	A	B	C	R^2
5	0.005	-0.9017	40.362	0.9213
10	0.0076	-1.4089	65.58	0.9185
15	0.0103	-1.9606	93.596	0.9909
20	0.0179	-3.4498	166.94	0.9509
25	0.0406	-7.5625	353.33	0.987

Table 3. Table of the modelled equation of the variation of resistance with firing temprature for various iron content for resistor length of 10mm

Composition(%Fe)	A	B	C	R^2
85	2E-05	-0.03	12.607	0.9093
90	2E-05	-0.034	12.893	0.927
95	1E-05	-0.0198	7.7045	0.9928

Table 4a. Table of the modelled equation of the variation of Normalized resistance with length for various iron compostion at firing temperature of 600°c

Composition(%Fe)	A	B	C	R^2
75	0.0028	-0.0372	0.1707	0.9922
85	o.oo4	-0.0769	0.3675	0.9447
95	0.0034	-0.0563	0.2621	0.9842
95	1E-05	-0.0198	7.7045	0.9928

Table 4b. Table of the modelled equation Variation of Normalized resistance with length for various temperature for 75% iron concentration.

Composition(%Fe)	A	B	C	R^2
300	0.0031	-0.0469	0.2088	0.9816
400	0.0023	-0.0209	0.0896	0.9883
500	0.0014	0.0014	0.0428	0.9842

Table 5a. Table of the modelled equation of the variation of resistance with length for various iron composition at firing temprature of 800°c

Composition(%Fe)	A	B	C	R^2
80	0.0052	-0.0527	0.432	0.9604
85	0.0045	-0.0421	0.286	0.9851
90	0.0047	-0.063	0.334	0.9948
95	0.0037	-0.0505	0.258	0.9992

Table 5b. Table of the modelled equation of the variation of resistance with length for various firing temprature at 95% Fe

Composition(%Fe)	A	B	C	R^2
500	0.0351	-0.4962	2.11	0.9953
600	0.0299	-0.5195	2.418	0.9705
700	0.0333	0.0047	0.058	0.9967
1000	0.0026	-0.0306	0.144	0.9972

Table 6a. Table of the modelled equation of the variation of resistance with paricle sizes. With 95% Fe and resistor length of 10mm at various firing temprature.

Temperature °c	A	B	C	R^2
300	-4E-06	0.0234	-2,8229	0.999
400	-2E-05	0.0233	-2.7343	0.959
500	-9E-06	0.0133	-1.5514	0.9795
600	3E-06	-0.0009	0.8651	0.9986
1000	1E-07	0.0015	0.3245	0.9253

Table 6b. Table of the modelled equation of the variation of Resistance against particle size for various iron composition with a resistor length of 10mm at 1000°c firing temprature

Composition %Fe	A	B	C	R^2
75	5E-06	0.0001	2.5231	0.9842
85	-1E-05	0.0164	-2.5717	0.969
95	-1E-06	0.003	-0.085	0.9669

Table 6c. Table of the modelled equation of the variation of reistance with temprature for different particles sizes with resistor length of 10mm and 85% Fe

Particle size	A	B	C	R^2
0-250	4E-05	-0.06	24.141	0.9735
250-400	6E-05	-0.0938	36.98	0.9529
400-500	6E-05	-0.097	38.832	0.9865
500-850	0.0002	-0.2465	93.816	0.9222

Table7a. Table of the modelled equation of thevariation of resistance with moulding pressure at various firing temprature with a resistor length of 15mm and 75% Fe

Temperature	A	B	C	R2
300	-0.409	-1.7732	6.9371	0.9701
400	0.4846	-3.774	6.317	0.9652
500	1.4192	-5.8954	6.0121	0.9388

Table 7b.Table of the modelled equation of thevariation of resistance with moulding pressure at various firing temprature with a resistor length of 15mm and 75% Fe

Temperature (°c)	A	B	C	R^2
600	23.097	-105.31	123.47	0.9969
700	-4.754	6.8232	16.806	0.9912
800	-2.8681	2.1636	13.802	0-977
900	4.6442	-24.842	34.463	0.9875
1000	-4.4276	10.351	2.5287	0.999

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