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Carbon Nanotubes Effect for Polymer Materials on Break Down Voltage

Loai S. Nasrat[1], Berlanty A. Iskander[2], Marina N. Kamel[3]
[1]Department of Electrical Power and Machines, Faculty of Engineering, Aswan University, Egypt
[2]Department of Materials, Faculty of Engineering, American University in Cairo, Egypt
[3]Maintenance Engineer at Marwa Company, Aswan, Egypt

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ABSTRACT

Epoxy resin composites reinforced to different types of carbon nano-particles have been fabricated. Carbon black (20, 30 and 40 wt. %), graphene (0.5 to 4 wt. %) and carbon nanotubes (CNT) (0.5 to 2 wt. %) were added with different weight percentages to epoxy. The dielectric strength of composites was tested in several conditions such as (dry, wet, low salinity and high salinity). The mechanical characterization showed that the nano-composite Polymer enhanced by using these particles in the tensile strength. Thermal gravimetric analysis shows effect of these nano-particles on the thermal structure of epoxy resin. Scanning Electron Microscopic test is used to characterize the dispersion of carbon nano-particles and to analysis the fractured parts in the nano scale.

Corresponding Author:

Marina N. Kamel,
Maintenance Engineer at Marwa Company,
Aswan, Egypt.
Email: eng.marina2013@gmail.com

1. INTRODUCTION

The electrical behavior for epoxy hybrid composites depends on the added filler. Therefore the filler contains two types with different particle size (filler). The main purpose of filler addition is the improvement of electrical performance and mechanical feature. The resistivity of composite with filler is maximized comparing with nano and micro composite [1]. Conducting polymers have been the subjects of study for many decades as possible synthetic metals. However, the practical uses of conducting polymers are not very likely because of their poor mechanical properties and process ability that rarely meet the industrial requirements. Thus, a unique combination of electronic and mechanical properties of composites of conducting polymers with conventional polymers seems to have great applications. The applications of conducting polymers are used in light emitting diode (LED) [2], sensors [3], a microbial fuel cell (MFC) [4], Organic Thin Film Transistor (OTFT) [5]. The field of nanoscience has blossomed over the last two decades and the importance of nanotechnology increase in areas such as computing, sensors, biomedical and many other applications. In this regard the discovery of grapheme [6] and graphene-based polymer nano composites is an important addition in the field of nano science. Carbon nanotubes were first discovered in 1991, and quickly became the focus of much research activity, due to their exceptional electrical, mechanical, and thermal properties [7].Carbon assumes an array of structural forms, such as diamond, graphite, graphene, fullerenes, carbon nanotubes, and amorphous carbon [8], [9]. Carbon nanomaterials have emerged as a rising star in the material science community, during the past two decades [10-12]. Carbon nanotubes and graphenes are also studied for the development of batteries, supercapacitors, and fuel cells [13-20]. Most of the research works on the conductive polymers composites focus on modification of the electrical properties

by subjecting their structures to various physical and chemical conditions to produce new systems with modified conductivity [21-24].

This paper aims to improve epoxy properties (electrical and mechanical) by adding fillers as CB, CNT and graphene. It focuses on trying to find an appropriate weight percentage composition of such composites in order to enhance the dielectric strength in different conditions. Also, some mechanical properties of the composites were investigated such as elongation at break and tensile strength. Soft program (Curve fitting) is used to interpret the Equation between dielectric strength values and different percentages of filler.

2. MATERIALS AND METHODS

2.1. Materials

The samples were prepared using epoxy polymer mixed with hardener 2 to 1 for 30min in vacuumed room. The fillers used in the present study are carbon black (CB), carbon nanotubes (CNT) and graphene (G) fillers.

2.2. Nano Composite Preparation

Epoxy nano composites were prepared in the laboratory by direct dispersion method following a protocol developed by one of the authors to get the best possible dispersion [25].

CNT are first dispersed in ethanol solution and a long hand. After completing evaporation of ethanol, then CNT is directly added to the epoxy resin with a well-done hand mixing until a complete dispersion is observed. Finally the hardener is added to the mixture.

Graphene is added to epoxy and then mixed well until the viscosity change then added hardener. The same way of preparation for CB is used for graphene. After preparing each sample, it is placed in the form of disc with diameter 5 cm and thickness 1 mm for dielectric strength test under pressure to reduce porosity forming during hardening. Also a very thin coat of silicon is added to disk to prevent the stickiness of epoxy composites to disk all samples are left about 24 to 36 hours in a well-ventilated room in order to be fully hardened. Table 1 shows the mixing formulation of epoxy with different fillers.

Table 1. Mixing formulation of epoxy with different fillers

Composite number	Filler added to epoxy	Filler wt.%
1	without filler	0
2	CNT	0.5%
3	CNT	1%
4	CNT	1.5%
5	CNT	2%
6	Graphene	0.5%
7	Graphene	1%
8	Graphene	1.5%
9	Graphene	2%
10	Graphene	3%
11	Graphene	4%
12	CB	20%
13	CB	30%
14	CB	40%

2.3. Dielectric Breakdown Strength Test

Dielectric strength of an insulating material is the maximum electric field strength that it can withstand intrinsically without breaking down, without experiencing failure of its insulating properties. It is expressed in voltage gradient items, such as voltage per thickness (kV/mm). It is one of the major electrical properties for insulation.

The failure is characterized by an excessive flow of current (arc) and by partial destruction of the material. Dielectric strength is measured through the thickness of the specimen (which is equal to 1mm), and is expressed in volts per unit of thickness. Sets of samples have been prepared and tested using A.C voltage according to IEC 60243 in different temperatures range. Samples are in the form of disc with diameter 5 cm and thickness 1 mm. For each test, the average result of five samples has been taken to minimize the error. Figure 1 shows the circuit used for dielectric break down strength test.

By using curve fitting methods, we can create access and modify curve fitting objects. That allowed to like plot and integrate, to perform operations that uniformly process the entirety of information

encapsulated in a curve fitting object. Dielectric properties were investigated at four conditions; dry, wet, low salinity (5% salt solution) and high salinity (10% salt solution).

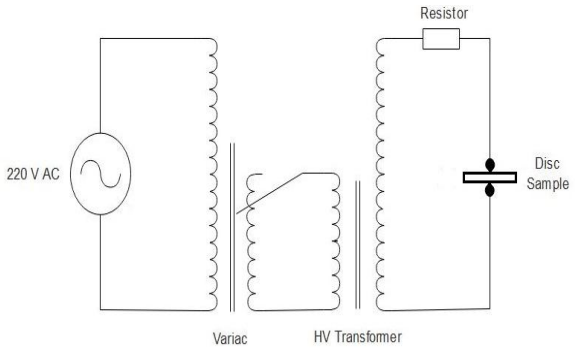


Figure 1. Schematic diagram for the dielectric strength testing circuit

2.4. Tensile Test

Mechanical test such as Tensile strength and percentage elongation at break are performed to illustrate the ability of samples to withstand the mechanical force. The dimensions of the sample are 5cm length and 1 mm thickness for tensile strength and 10 cm length and 1 mm thickness for Elongation at break. All the samples with certain weight percentage addition were tested three times with an average result calculated.

2.5. Morphology Test

Morphology test such as scanning electron microscope (SEM-JOEL JSM-840) is conducted by using a beam of electrons to create an image of the specimen. For SEM examinations, the epoxy samples were first coated with gold in a vacuum oven. The JSM-840 examines structure by bombarding the specimen with a scanning beam of electrons and collecting slow moving secondary electrons that the specimen generates. These are collected, amplified, and displayed on a cathode ray tube although now, most are driven by PCs and these computer-generated images are displayed on LCDs.

2.6. Thermo-Gravimetric Analysis (TGA) Test

Thermo-gravimetrical analysis is an instrument simply measures weight change vs. temperature. It is very effective technique to study chemical and physical phenomena as a function of temperature. For TGA measurement, samples were cut as small pieces (10 mg). The sample is heated from 0 to 500° C at a constant rise of temperature (10° C/min) in nitrogen atmosphere, while the sample weight is continuously monitored by computer screen.

3. RESULTS AND ANALYSIS

3.1. Electrical Results

3.1.1. Dielectric Breakdown Strength of CB/ Epoxy Composites

The dielectric strength for epoxy has been studied with different filler wt. % in different conditions: dry, wet, low salinity and high salinity.

Comparison between percentages of CB/Epoxy leads to 20%wt CB the maximum value in these composites. It reaches to 21.31 kV at dry condition, 25.19 kV at wet condition, 16.62 kV at low salinity condition and 15.12kV at high salinity condition.

Figure 2 shows the dielectric breakdown strength of CB/Epoxy composites in different conditions

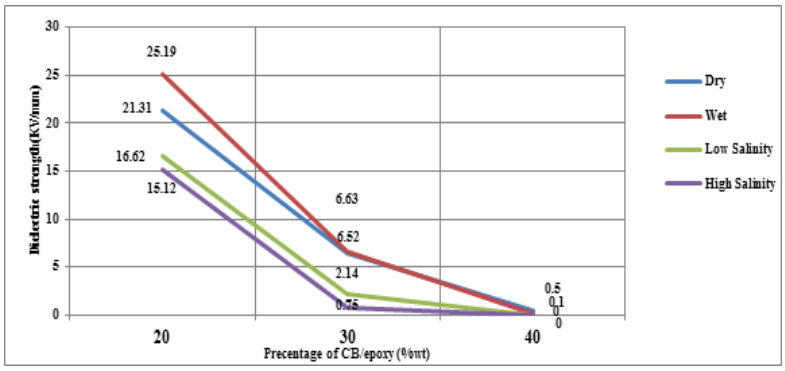


Figure 2. Dielectric breakdown strength of CB/Epoxy composites in different conditions

3.1.2. Dielectric Breakdown Strength of CNT/Epoxy Composites

Figure 3 shows that 1%wt CNT has the maximum value of dielectric strength. It reaches to 3.5 kV at dry condition, 2 kV at wet condition, 0.7 kV at low salinity condition and 0.2 kV at high salinity condition.

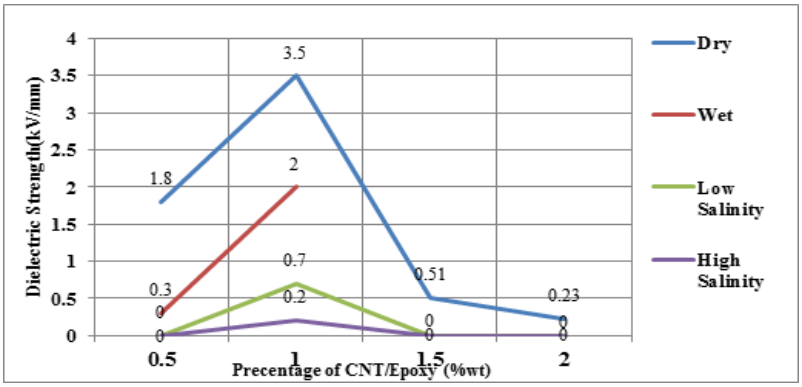


Figure 3. Dielectric breakdown strength of CNT/ Epoxy composites in different conditions

3.1.3 Dielectric breakdown strength of G/Epoxy composites

Figure 4 explains that, 3%wt G has the maximum value of dielectric strength. It reaches to 30.17 kV at dry condition, 27.46 kV at wet condition, 24.46 kV at low salinity condition and 22.63 kV at high salinity condition.

There are no readings of dielectric strength for samples in CB/Epoxy and CNT/Epoxy because these samples become conducting material.

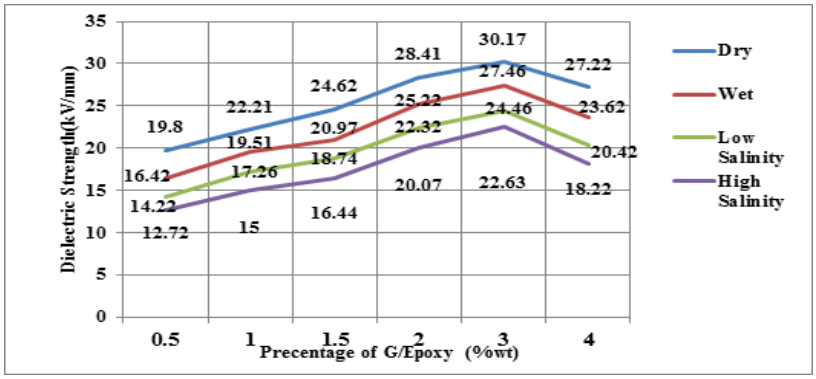


Figure 4. Dielectric breakdown strength of G/ Epoxy composites in different conditions

3.1.4. Dielectric Breakdown Strength of epoxy blank

At dry condition, epoxy has the maximum value of dielectric strength (35.09 kV/mm), while other samples have the minimum value of dielectric strength. At wet, low salinity and high salinity conditions, dielectric strength is improved by increasing fillers percentage to epoxy.

The dispersion of filler particles is important factors for controlling of breakdown voltage [26], so from Figure 7(B), dielectric strength for CB is low.

Nanocomposite exhibits higher breakdown strength because of the tight intimacy between the nano filler and epoxy resin. However, micro fillers have a comparatively small ratio surface compared with nano fillers which forms a loss bond with substrate and can not remove defects as nano fillers does but instead introduce more defects that lower breakdown strength.

Breakdown strength is improved when nano filler is added to the epoxy resin, while the breakdown strength of epoxy resin lowers when micro filler is added. The breakdown strength of nanocomposite is enhanced with the increasing content of nano filler [27]. Figure 5 shows the dielectric breakdown strength of epoxy blank in different conditions.

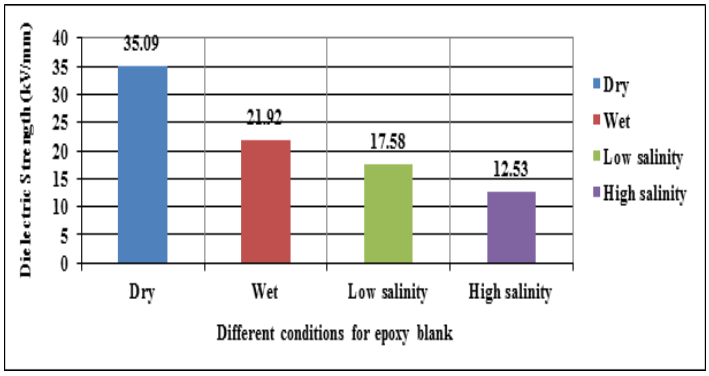


Figure 5. Dielectric breakdown strength of epoxy blank in different conditions

3.2. Soft Program Results

Curve fitting (regression analysis) is used to find the "best fit" line or curve for a series of data points. Most of the time, the curve fit will produce an Equation that can be used to find points anywhere along.

Figures 2- 4 show that:

1- Equations of curve fitting results for the dielectric strength of composites under dry condition are:

$$y = 0.0438x2 - 2.7945x + 44.87 \quad (1)$$

FIGURE $y = 9.8667x3 - 38.98x2 + 44.603x - 11.99$ (2)

$$y = 0.344x4 - 3.6208x3 + 11.092x2 - 6.8905x + 20.974 \quad (3)$$

2- Equations of curve fitting results for the dielectric strength of composites under wet condition are:

$$y = 0.0601x2 - 3.6605x + 55.78 \quad (4)$$

$$y = 3.4x - 1.4 \quad (5)$$

$$y = 0.0345x4 - 1.0258x3 + 3.893x2 + 0.694x + 15.354 \quad (6)$$

3- Equations of curve fitting results for the dielectric strength of composites under low salinity condition are:

$$y = 0.0617x2 - 3.299x + 43.44 \quad (7)$$

$$y = 2.8x3 - 11.2x2 + 13.3x - 4.2 \quad (8)$$

$$y = -0.069x4 - 0.0456x3 + 0.8001x2 + 4.0132x + 12.123 \quad (9)$$

4- Equations of curve fitting results for the dielectric strength of composites under high salinity condition are:

$$y = 0.0681x2 - 3.48x + 43.11 \quad (10)$$

$$y = 0.8x3 - 3.2x2 + 3.8x - 1.2 \quad (11)$$

$$y = -0.0327x4 - 0.5908x3 + 3.3465x2 - 0.343x + 12.222 \quad (12)$$

Parameter Y can be represented value of dielectric strength (kV/mm) at all different conditions. Parameter X varies to different fillers such as CB, CNT and G. Equations (1), (4), (10) show CB, Equations (2), (5), (11) explain CNT and Equations (3), (6), (12) explicate G.

3.3. Mechanical Results

Tensile strength tests (TS) are carried out in order to illustrate the ability of samples to withstand the mechanical forces.

Table 2: Tensile properties of composites with different percentage of fillers

T	Tensile strength (MPa)	Tensile strain at break %	Tensile extension at break (mm)
Epoxy Blank	14	43.23	17.29
20% CB	15	5.94	2.37
30% CB	22.3	4.69	2.7
40% CB	23.3	4.06	1.625
1%CNT	28	3.12	1.24
2%CNT	25.6	2.63	1.053
1% G	22.3	5.36	2.14
2% G	30	4.05	1.62
3% G	38	1.43	0.58

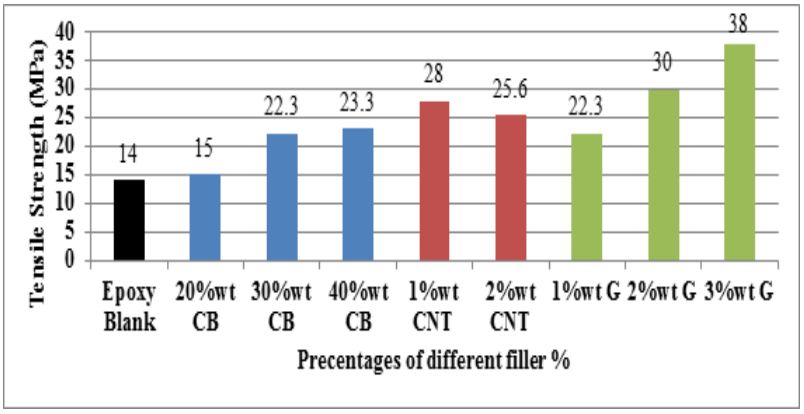
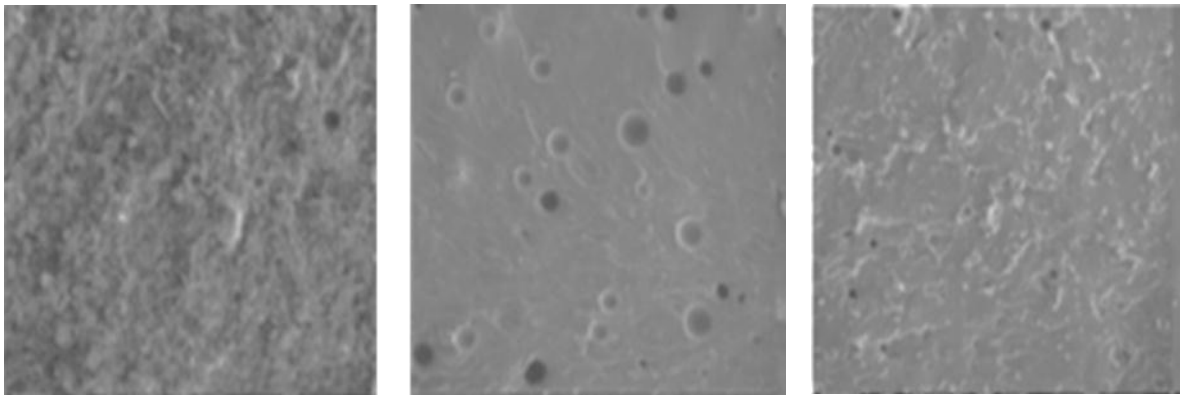


Figure 6. Tensile strength of various composites

Table 2 and Figure 6 show that, tensile strength is inversely proportional to the increase of filler concentration. 3wt %G has the better tensile strength (38 MPa) than all samples. 40wt %CB and 1wt %CNT have the better tensile strength than pure epoxy. The worst concentration is 2wt %CNT.

3.4. Morphology Results

SEM test has been studied four various magnifications (150x, 1500x, 6000x and20000x) were used to examine three different samples (40%CB, 1% CNT and 1% G).



(a) (b) (c)

Figure 7. SEM photographs of the (a) 40% carbon black, (b) 1% CNT and (c) 1% G of sample

Figure 7 (a) shows that air bubbles are existed at big form. Figure 7 (b), an agglomeration of several carbon nanotubes is observed on the fractured surface of the Epoxy/CNT composite. Figure 7 (c) shows that the agglomeration is less than other samples, so mechanical properties are improved.

3.5. Thermo-Gravimetrical Analysis (TGA) Measurements

Figure 8 illustrates a comparison between the changes in samples weight percentage over the increase of the temperature.

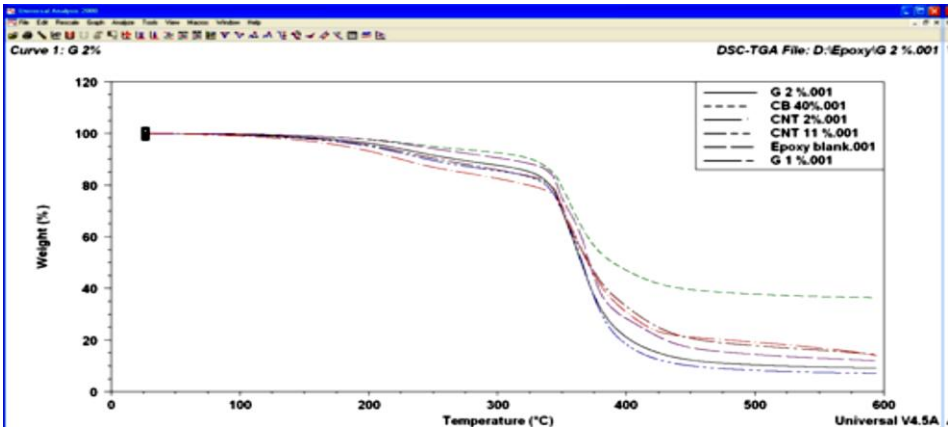


Figure 8. Percentage of thermal stability of epoxy composites with different percentage of fillers

Figure 8 shows that TGA test illustrate that 40wt. %CB represent the best sample from stability point, and epoxy without any filler represents the worst sample.

All specimens started to decompose around 330 °C and completed decomposition around 445°C. The residual mass of pure epoxy is about 11.9 wt. % at 593 °C, 40%wt CB is calculated to be 36.477wt. %, 1 and 2 wt. % CNT/Epoxy are approximately 12.79 and 13.92wt and 1 and 2 wt. % G/Epoxy approximately 12.09 and 13.34 wt. %, respectively. Weight loss of different nano carbon sample is the smallest of all samples, so TGA test represents the best sample from thermal stability point and the sample from thermal stability point.

Table 3. TGA for epoxy composites with different percentage.

Sample	Start temperature (C)	Stop temperature	Change %
Epoxy Blank	328.22	449.35	70.73
40% CB	314.04	425.58	49.75
1% CNT	325.93	442.49	72.4
2% CNT	340.56	435.64	55.25
1% G	331.42	440.67	70.77
2% G	338.27	447.07	59.84
3% G	293.07	499.26	70.461

4. CONCLUSION

It can be concluded from the expermintal work that,

a. The dielectric strength is improved by increasing of G percentage in the samples. [3%wt G/epoxy] sample have the highest dielectric strength of:
   1) 30.17 kV/mm (52.37% improvement) under dry condition,
   2) 27.46 kV/mm (67.24% improvement) under wet condition,
   3) 24.46 kV/mm (72% improvement) under low salinity condition and
   4) 22.63 kV/mm (78% improvement) under high salinity condition.
b. Increasing fillers to epoxy enhance the mechanical properties; 3wt. % G has the maximum value of tensile strength (38MPa).
c. The homogeneity of the composite is useful for mechanical behavior of the nano composites
d. TGA results indicated that the addition of carbon nanoparticles to the epoxy resin doesn't affect the thermal stability of the epoxy resin which is a good behavior for the carbon particles.

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BIOGRAPHIES OF AUTHORS

Dr. Loai Nasrat is a professor at Aswan University, Faculty of Eng., Elect. P&M Department. He has been actively involved in both basic and applied research in the area of HVengineering. His research interests include HV insulators, polymeric materials, HV cables insulations and environmental studies. Author and co-author of more than 60 papers on HV polymeric insulating materials, published in technical journals and proceedings of national and international conferences.
loaisaad@yahoo.com

Dr. Berlanty Iskander is a Professor in Materials and production engineering at Akhbar el yom Academy and Adjunct at American University in Cairo. She has actively involved in both preparation in material and applied research in testing and characterization of materials Author and co-author in more than 35 papers. External Examiner for Masters and Doctorates Degree at Banha university. Helwan University and Mansora university. Reviewer for many research paper in Local and international Journals.
berisk@aucegypt.edu

Marina Nader Kamel was born in Aswan, Egypt on 2nd February 1991. She received the B.Sc. (FEE) Electrical Engineering degree in 2013 from Faculty of Energy Engineering, Aswan, Egypt and currently pursuing “master of Electrical Engineering” from Faculty of Engineering, Aswan University, Egypt. She is working at Marwa Company in Aswan, Egypt as a Maintenance Engineer.
eng.marina2013@gmail.com