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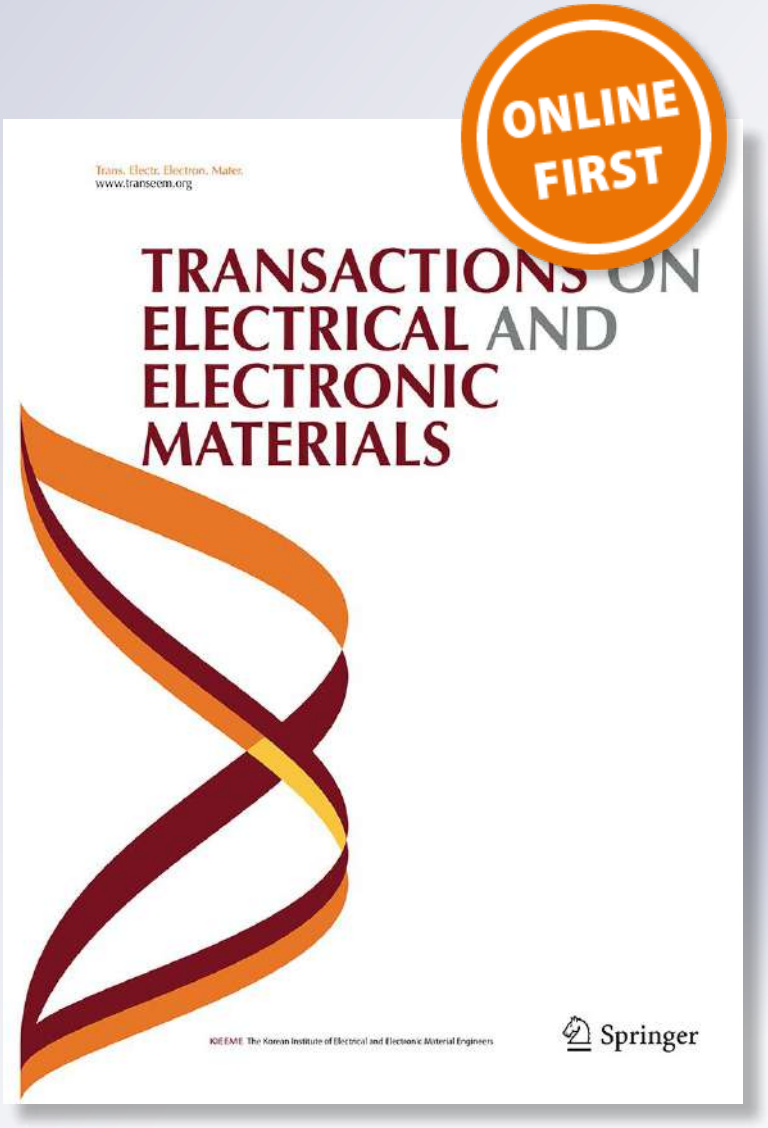

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REGULAR PAPER

Synthesis and Characterization of Flexible Resistive Humidity Sensors Based on PVA/PEO/CuO Nanocomposites

Ahmed Hashim[1] · **Yahya Al-Khafaji**[2] · **Aseel Hadi**[3]

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Abstract
Synthesis of polyvinyl alcohol (PVA)–polyethylene oxide (PEO)–copper oxide (CuO) nanocomposites and studying their structural and optical properties for humidity sensors applications were investigated. The prepared humidity sensors have lightweight, low cost, flexible and high sensitivity compare with other humidity sensors. The results showed that the nanocomposites have high absorption in UV region. The absorbance of (PVA–PEO) blend increases with increase in CuO nanoparticles concentrations which may be used for solar cell, transistors, diodes and other electronic applications. The optical constants increase while the transmittance and energy gap decrease as CuO nanoparticles concentrations increase. The results of application showed that the (PVA–PEO–CuO) nanocomposites with different copper oxide nanoparticles concentrations have high sensitivity for relative humidity which may be used as sensors for different humidity ranges.

Keywords Sensitivity · Optical properties · Nanocomposites · Blend · Humidity · Copper oxide

1 Introduction

Humidity sensors have gained advanced applications in industrial processing and environmental control. For manufacturing, highly sophisticated integrated circuits in semiconductor industry, humidity levels are constantly monitored in wafer processing. There are many domestic applications, such as intelligent control of the living environment in buildings, cooking control for microwave ovens, and intelligent controller. In automobile industry, humidity sensors are used in rear window defoggers and motor assembly lines. In medical field, humidity sensors are used in respiratory equipment, sterilizers, pharmaceutical processing, and biological products. In agriculture, humidity sensors are used for green-house airconditioning, plantation protection (dew prevention), soil moisture monitoring. In general industry, humidity sensors are used for humidity control in chemical gas purification, dryers, ovens, film desiccation, paper and textile production, and food processing. Humidity sensor plays an important role in every part of the Earth and automated industrial processes. To have a desirable surrounding atmosphere, it is essential to monitor, detect and control the ambient humidity under different weather ranging from low temperature to high temperature or in mixtures with other gases by precise and provident sensors [1]. Humidity measurement is one of the most significant issues in various areas of applications such as agriculture, climatology. In recent years improvements in sensor manufacturing technologies have taken place in high-speed, low-power and low-cost microelectronic hybrid circuits, modern signal conditioning methods and advances in miniaturization technologies. Utilization in intelligent systems and networks as monitoring sensors to determine the soil moisture during irrigation in agriculture, or for diagnosis of corrosion and erosion in advanced batteries, like lithium batteries, are among the applications of humidity sensors. Nanocomposites incorporating ceramics, ceramics/polymers and polymers/carbon nanotubes with nanoporous, nanofiber and nanowire forms are amongst the most promising materials for future applications [2].

Moisture sensing is crucial for numerous applications and coming technologies. Therefore, there has been an rising developing materials of demand for high show sensors

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of humidity in manufacturing production, national security, environmental monitoring, etc. In excess of the years, diverse sensing materials, containing semiconducting materials, macromolecule compounds, carbon nitride film porous ceramics, and porous silicon have been explored to distinguish humidity. However, the humidity sensors production with these materials consequences in fewer reliability and stability, and from now on limits the humidity sensors development for the practical applications. The material of sensing, used for the humidity sensor, should adsorb the moisture efficiently. Now, numerous carbon materials have exhibited very good of molecular adsorption properties [3].There are great materials numbers, which have been examined and used like elements of sensing in moisture measuring devices. In the middle of them, ceramic oxides have revealed advantages in thermal, physical and mechanical strength. Ceramic porous sintered bodies have mostly been used in humidity sensing. Monitoring of surface activity and porosity acts extremely important role in measuring the moisture responsive electrical behavior of ceramic products [4]. Humidity sensors is attracting attention due to their applications in medical, construction, food processing industries, and meteorological. Conventionally, there are two methods used to determine relative humidity, by measuring exchanges in changes in luminescence features. or oscillation frequency. The chemical reaction between the material surface and water vapor lead to change in resistance and capacitance of moisture sensors. the nano-structured materials possing large surface area give high response as compared with thin film and bulk materials when they used in sensors applications. In fact, humidity sensors based onnanorods, nanowires, nanocomposites, nanotubes have each and every one been established [5]. Poly(vinyl alcohol) is soluble polymer in water that has involved exacting interest due to its biocompatibility and hydrophilicity properties. PVA is harmless and has excellent thermal stability, creation it a hopeful candidate to be used in biotechnology and biomedicine fields [6]. Poly(ethylene oxide) has attracted attention in recent years due to their solubility in water, biodegradability, non-toxicity, biocompatibility. It has numerous applications, such as agricultural films, paper coating, textile fibers, and electronic devices [7]. Basically, polymer metal oxide is used in humidity sensor applications and is prepared by conventional and advanced wet chemical processing methods at room temperature and is mainly developed to offer porous bodies. The advantage of an absorbent spongiform surface rather than a condensate is a greater permeability of water molecules, so water vapour molecules can easily pass through the pore openings and capillary condensation occurs in the capillary porous structures which are formed between the grain distributions in ceramic surface during the pore removal process [8]. CuO has attracted much attention. CuO is a p-type semiconductor material with small band gap energy of only 1.2 eV at room temperature. It has been shown that CuO can be used in high temperature high temperature superconductors, gas sensor, magnetic storage media, catalysis and field emitters [9]. The nanocomposites have different applications in many fields such as pressure sensor [10, 11], antibacterial [12, 16], energy storage [17–19], … etc. In this paper, preparation of (PVA–PEO–CuO) nanocomposites and studying their structural and optical properties for humidity sensors have low weight, low cost, easy fabrication, flexible and high sensitivity compare with other types of humidity sensors.

2 Materials and Methods

The nanocomposites films of polyvinyl alcohol- polyethylene oxide and copper oxide nanoparticles were synthesized by using casting method. The copper oxide (CuO) nanoparticles was obtained as powder from US Research Nanomaterials, Inc, USA with size (25–55) nm, crystalline and high purity (99.95%). The 1 g of polyvinyl alcohol and polyethylene oxide were used to prepare the films by dissolving them in 30 ml of distilled water, the weight percent (85 wt% PVA and 15 wt% PEO). The solution was stirred to achieve homogeneous solution. Different concentrations of copper oxide nanoparticles were added to polymer blend (5, 10 and 15) wt%. The samples were prepared with thickness range (29–35) μm where the thickness were measured by digital micrometer. The double beam spectrophotometer is used to measure the optical properties of (PVA–PEO–CuO) nanocomposites in wavelength (280–800) nm. Glass slides was employed to prepare samples for humidity sensor application by dropping of synthesized nanocomposites solution on glass. The steam of water was used as a source of humidity to apply on each sample. The network of control observed and monitored variations in the humidity. The electrical resistance of nanocomposites for humidity in range (30–70) was measured via employing Keithley electrometer type 2400.

The absorption coefficient (α) of nanocomposites can be determined by [20, 21].

$$\alpha = (2.303A)/t \tag{1}$$

A and t refer to absorbance and thickness of sample respectively, while the non-direct transition model for (PVA–PEO–CuO) nanocomposites is described by [22]:

$$\alpha h\upsilon = B(h\upsilon - E_g)^r \tag{2}$$

where B is constant, E_g is photon energy. The relation between The refractive index (n) and the reflectance (R) is given Eq. (3) [23]:

$$n = (1 + R^{1/2})/(1 - R^{1/2}) \tag{3}$$

The extinction coefficient (k) is defined by the equation [24]:

$$K = \alpha\lambda/4\pi \tag{4}$$

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While Eqs. (5) and (6) are employed to calculate the real (ε_1) and imaginary (ε_2) parts of dielectric constant of prepared nanocomposites [25, 26]:

$$\varepsilon_1 = n^2 - k^2 \tag{5}$$

$$\varepsilon_2 = 2nk \tag{6}$$

The optical conductivity of (PVA–PEO–CuO) can be determined by following equation [27]:

$$\sigma = \frac{\alpha nc}{4\pi} \tag{7}$$

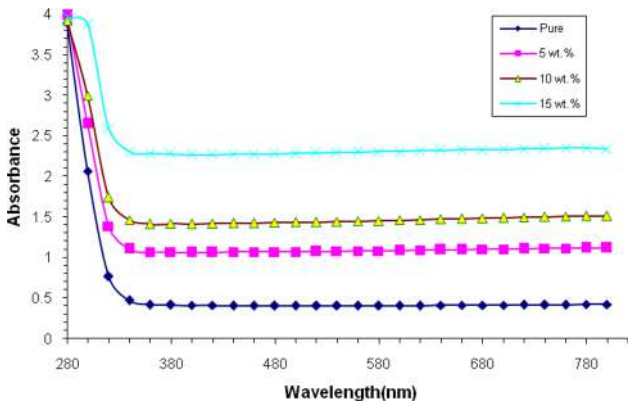

Fig. 1 Variation of absorbance of (PVA–PEO–CuO) nanocomposites with wavelength

3 Results and Discussion

Figure 1 shows the variation of absorbance for (PVA–PEO–CuO) nanocomposites with wavelength. As shown in Fig. 1, the absorbance was increased in the UV region. This not only, the photon energy enough to interact with atoms, but also, the donor has electrons level near to conduction band at UV energy. The nature of transition of possible electron can change when the transmitted and absorbed radiation changes. The observed absorbance in UV region was higher than that of the visible and near infrared regions, and the main reason is photon energy insufficient to interact with atoms, therefore, transmitting of photons will occur [28]. The absorbance rises with increase in copper oxide concentrations which is due to the agglomeration of CuO nanoparticles as shown in Fig. 2 which is show that the arrangements of CuO nanoparticles in (PVA–PEO) polymer matrix at magnification power (10×). The figure show that the CuO nanoparticles is aggregated as a clusters at lower concentrations. When increasing the concentrations of CuO nanoparticles, the nanoparticles form a paths network inside the (PVA–PEO) blend, hence the charge carriers are allowed to pass during the routes [29, 30]. Figures 3 and 4 represent the energy band gap for allowed and forbidden indirect transitions of nanocomposites. Both figures show the energies gaps of (PVA–PEO–CuO) nanocomposites reduce with increase the CuO nanoparticles concentrations, It is worth to note that this performance caused by creating of levels

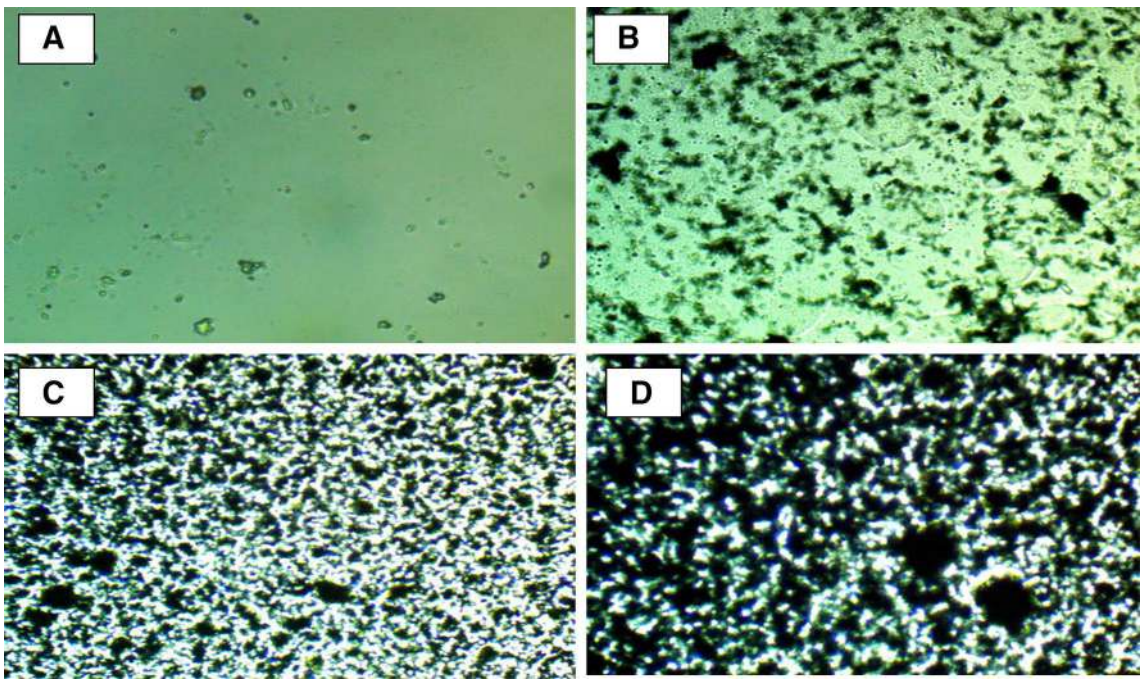

Fig. 2 Microscope images (×10) for (PVA–PEO–CuO) nanocomposites: **a** for blend **b** for 5 wt% CuO, **c** for 10 wt%CuO **d** for 15 wt%CuO

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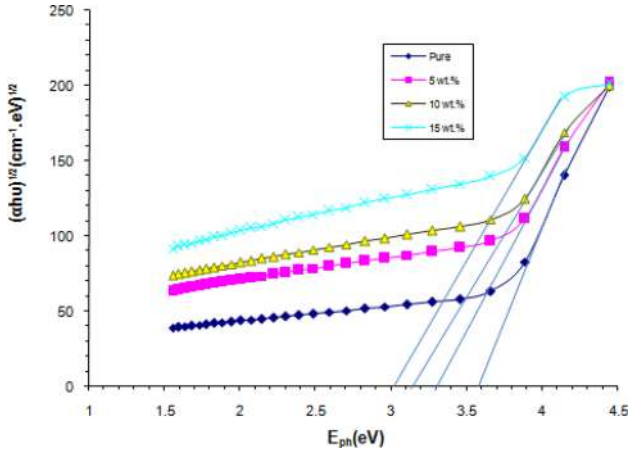

Fig. 3 Energy gap for transitions of allowed indirect for (PVA–PEO–CuO) nanocomposites

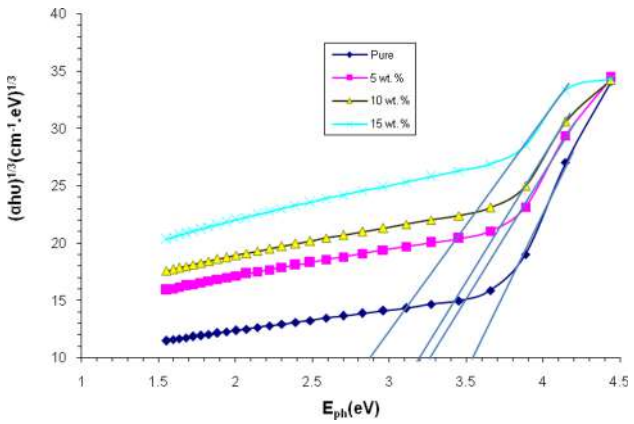

Fig. 4 Energy gap for transitions of forbidden indirect for (PVA–PEO–CuO) nanocomposites

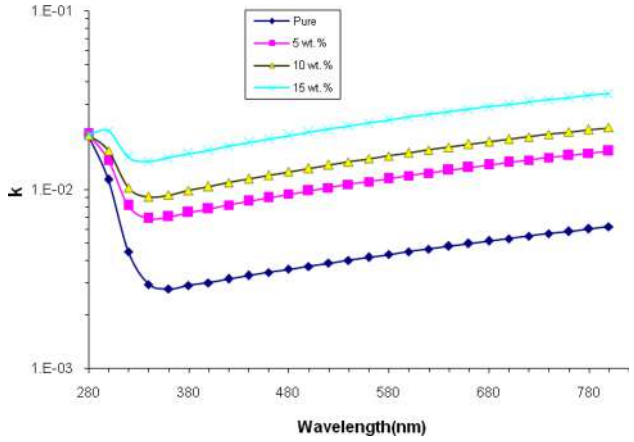

Fig. 5 Variation of extinction coefficientwith wavelength

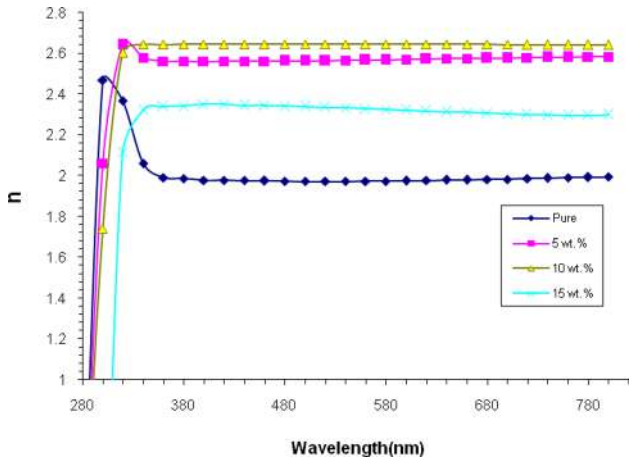

Fig. 6 Refractive index of (PVA–PEO–CuO) nanocomposites as a function of wavelength

in the energy gap; there are two stages when the electron transition in this case, first stage is occupy the transition in the valence band and second stage is the local levels and to the conduction band as consequence of increase the concentrations of CuO nanoparticles; the conduction of electronic rely on the concentrations of CuO nanoparticles. It is clear from the results the presence of strong intermolecular interaction between composite and dopant component this lead to reduce the distance between anti-bonding and bonding molecular orbitals and a lesser photon energy is required to expect electrons from π to π* molecular levels was reduced as well [31].

Figure 5 shows the variation of extinction coefficient for (PVA–PEO–CuO) nanocomposites as a function of photon wavelength. The increase in the concentrations of CuO nanoparticles lead to increase in the extinction coefficient of nanocomposites, this because of CuO dispersion in the (PVA–PEO) blend. The extinction coefficient of (PVA–PEO–CuO) nanocomposites has higher values at UV region which is related to the higher absorbance. The refractive index of (PVA–PEO–CuO) nanocomposites with wavelength is shown in Fig. 6. The refractive index of (PVA–PEO–CuO) nanocomposites increases with an increase the concentrations of CuO nanoparticles; it is reduced with an increase of the photon wavelength which is may be attributed to the increase of nanocomposites density [31].

The dielectric constant (real and imaginary parts) of (PVA–PEO–CuO) nanocomposites are shown in Figs. 7 and 8. From these figures it is clear that high concentration of CuO leads to increase of absorption coefficient and refractive index and subsequently lead to increase of the real and imaginary parts of dielectric constant of (PVA–PEO) [32].

Figure 9 shows the optical conductivity of (PVA–PEO–CuO) nanocomposites with different concentrations of CuO nanoparticles. It is clear that is affected by

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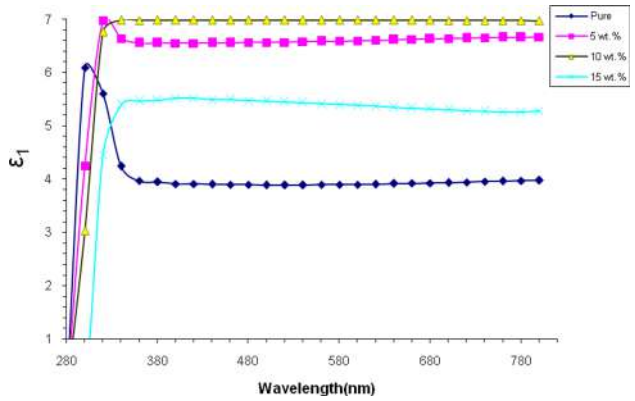

Fig. 7 Real part of dielectric constant of (PVA–PEO–CuO) nanocomposites

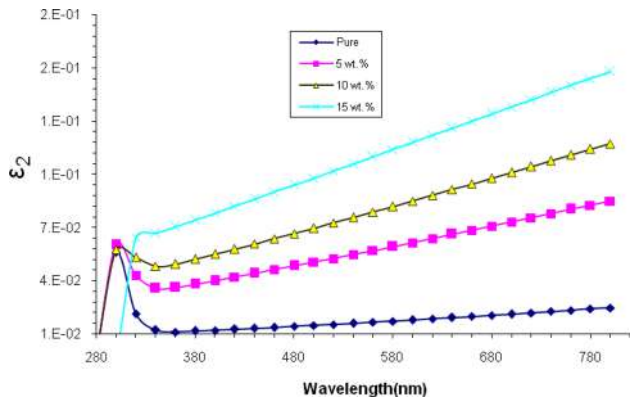

Fig. 8 Imaginary part of dielectric constant of (PVA–PEO–CuO) nanocomposites

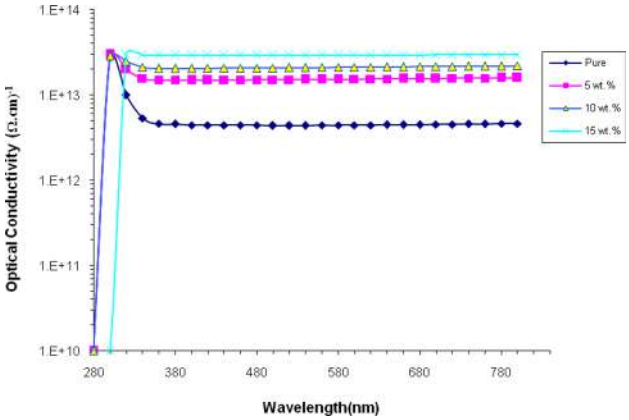

Fig. 9 Optical conductivity of (PVA–PEO–CuO) samples with concentrations ofCuO nanoparticles

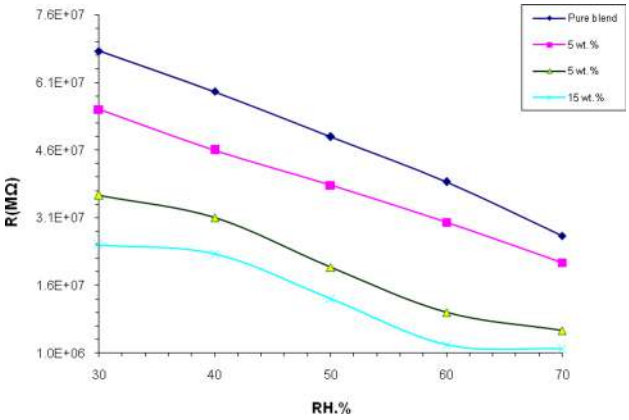

Fig. 10 Variation of resistance for (PVA–PEO–CuO) nanocomposites with humidity

increasing in CuO nanoparticles concentrations accompanying the rise in the refractive index and absorption coefficient [33].

Figure 10 shows the variation of electrical resistance with relative humidity for (PVA–PEO–CuO) nanocomposites. The results showed that there is an inverse behavior between relative humidity and resistance for all samples. Also, There is a proportional relationship between nanocomposites sensitivity and humidity, the reason behind this is: PVA and PEO mixed with water illustrate higher sensitivity at moisture sensing at (r.t). Adsorption the humidity on nanocomposites surface lead to decease it resistance in fact, the reason is increasing the charge carriers. Subsequently lead to dissociate the hydrogen ion on the (PVA–PEO–CuO) nanocomposites surface. the hydroxyl groups are formed as a result of bonded between $[H]^+$ with surface lattice of oxygen atom according to the following equation [34]:

$$H^+ + O_0 \leftrightarrow [OH]^- \tag{8}$$

where O_0 refer to oxygen atom relate with $[H]^+$. Then free electrons are released as a result of bonded between the hydroxyl groups and the atoms lattice. This electron is responsible for electrical conduction [34–36].

All the samples showed much lower resistance with higher relative humidity, showing that the conduction happened mainly at the surface of grain, No doubt this is because absorbed water molecules. The water absorption lead to increasing of humidity rate, which is relate with changing in the resistance. Chemisorption may happen at low humidity rates, because there are two surface formed of hydroxyls with the charge transport occurrence by the mechanism. As compared with high humidity rates, physical adsorption happen on a top of the chemisorbed layer by water. The chemisorbs occurred on the grain where found water molecules at low moister lead to form hydroxyl groups as carriers to the charge. Then primary water molecules are adsorbed, follow by water molecules bonded with hydrogen atom to form H_3O^+, and the dominant surface charge carriers

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will be H_3O^+. When still additional water is adsorbed, the molecules of water clustering takes place, forming a film of liquid-like multilayer of hydrogen bonded molecules of water, where each water molecule is only alone bonded to a hydroxyl group. Since dissociation of H_3O^+ into H_2O and H^+ is actively favourable in H_2O, where proton is the dominant positive charge transferor in high humidity environment [37–39]. It is well famous that vapor water adsorption on the CuO nanoparticles surface leads to alter of the electric circuit impedance. The high porosity in CuO nanoparticles surface, the additional H_2O molecules was adsorbed and it is worth to say high performance of employed sensor is achieved. Protonation and deprotonation of surface hydrolysis cause the electrical conductivity of cupric oxide. On the other hand the moisture adsorption within CuO shells can be explained as: humidity adsorption affects the protonic conduction on the surface and conductivity changes with the quantity of water being adsorbed. At the low humidity, conduction is due to hopping of proton between hydroxyl ions on first layer of chemisorbed water, while at high rate of the humidities, protons hop between physisorbed molecules with a Grotthus chain reaction mechanism [40]. The mechanism of the sensing may be attributed to the following probabilities: (1) At low RH mobility of CuO ions in the composite is restricted due to curling up of polymer chains. As humidity increases, polymer chain uncurls and becomes aligned by absorbing water molecules paving way for faster hopping of charge carriers, resulting in increased sensing response of the composite. (2) Porosity of the polymers may facilitate absorption of water molecules as RH increases causing a decrease in the resistivity of the nanocomposite [41]. In addition, on the other hand, in this paper, the copper oxide (CuO) nanoparticles which are used in this paper have a nanoparticle size (25–55) nm. The nano-sized grains and the high specific surface area also play an important role in the sensor performance. The samples show nano-scaled particles which leads to much more grain boundaries which result in more active sites available for water molecules to react. The smaller particle size which provided more grain boundary, more surface area and also more nanopores which offered more active sites for reacting with water molecules The water molecules are easily absorbed on the surface of the sensors due to the large surface of nanomaterials, which can improve the response and recovery characteristics [42–46].

4 Conclusions

In this paper the structural and optical properties of (PVA–PEO) blend and (PVA–PEO–CuO) nanocomposites as humidity sensors were studied. We have found that the absorbance of (PVA–PEO) blend increases with increase in CuO nanoparticles concentrations which may be used for solar cell, transistors, diodes and other electronic applications. The optical constants increase while the transmittance and energy gap decrease as CuO nanoparticles concentrations increase. The (PVA–PEO–CuO) nanocomposites have high absorbance in a high photon energy (UV-region) which can be used for solar cell application. The (PVA–PEO–CuO) nanocomposites have high sensitivity for relative humidity with low cost, low weight, high elastic and high corrosion resistance. The sensitivity values ranged from 60.2% for pure blend to 92.4% for 15 wt% CuO nanoparticles at room temperature. Also the electrical resistance of nanocomposites decreases with an increase the CuO and relative humidity.

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