



Fabrication and Characterization of (PVA-TiO₂)_{1-x}/ SiC_x Nanocomposites for Biomedical Applications



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THEORETICAL and experimental studies on structural, electrical and electronic properties of (PVA-TiO₂-SiC)_x nanocomposites for antibacterial application have been investigated with low cost, low weight and high activity for antibacterial. The (PVA-TiO₂-SiC) nanocomposites were prepared with different weight percentages of Silicon Carbide (SiC) nanoparticles. Electrical properties of (PVA-TiO₂-SiC) nanocomposites were studied with different temperature range. The experimental results showed that the conductivity, (PVA-TiO₂-SiC)_x nanocomposites is increased with an increase the weight percentages of Silicon Carbide (SiC) nanoparticles. The activation energy of (PVA-TiO₂-SiC)_x nanocomposites decreases with increase the weight percentages of Silicon Carbide nanoparticles. The total energies of the (PVA-TiO₂-SiC) nanocomposites were studied by using Gaussian 09(G09) program and density functional theory (DFT) with B3LYP/6-31G) basis set, and found that the total energies decreases with the increase the number of atoms forming the nanocomposites. The (PVA-TiO₂-SiC) nanocomposites tested for antibacterial applications. The results showed that the prepared nanocomposites have good antibacterial activity.

Keywords: Nanocomposites, Conductivity, Activation energy, Antibacterial, Silicon carbide.

Introduction

The production of novel polymeric nanocomposites have been widely studied by the current applications on nanosized inorganic fillers in the food packaging field, barrier applications, sensors, antimicrobial, conductive, coatings, antiballistic products and other materials. The new generation of these polymer nanocomposites contains nitrides (TiN, CrN, ZrN, BN), carbides (WC, SiC, B₄C), borides (WB, ZrB₂, TiB₂, CrB₂), metal oxides (Al₂O₃, Fe₃O₂, MgO, CeO₂, TiO₂, Y₂O₃, SiO₂, ZrO₂), carbon nanotubes, cellulose nanofibrils, and other kind of nanoparticles as disperse phase. The characteristics and properties

of the nanocomposites are influenced by the type of filler used as well as by the polymeric matrix. The applications encompass several areas such as electronic, medicine, military, aerospace, marine and vehicles [1]. It is expected that some organic inorganic nanocomposites will have synergetic behavior of their organic and inorganic components, and therefore, potential applications of these nanocomposites in optoelectronic devices, sensors, electromagnetic interference shielding etc [2]. Titanium dioxide (TiO₂) is an excellent photo catalyst material, which shows most promising prospect in solar energy conversion. This is due to several properties such as chemical stability, nontoxic, highly oxidative photo

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generated holes, highenergy conversion, highly transparent and high refractive index[3]. Silicon carbide nanostructures have specific properties useful for applications in microelectronics and optoelectronics. Actually, SiC has selected due to their properties as a high hardness, semiconductor processing equipment, etc. These characteristics make SiC a perfect possibility for high-power electronic devices. Silicon carbide nanocomposites have different advantages as far as their performance for use as special structural materials attribute to their excellent properties. This is accepted to be caused by discouraged non-radiative recombination in the confined clusters. SiC nanostructures have a great wonderful property which searches useful for applications in microelectronics and optoelectronics [4]. Among different polymers, poly(vinyl alcohol) (PVA) attracted the attention of researchers due to its optical characteristics, physical properties, film forming and biocompatibility. Furthermore, its properties still appear to be unique in terms of the price/performance ratio. To further enhance the properties, the incorporation of inorganic material is advantageous for forming a composite which could be lightweight, flexible and exhibit good moldability [5-7]. Applying nanotechnology for treatment, monitoring, control of diseases and diagnosis has been referred to as nanomedicine. The new resistant strains development of bacteria to current antibiotics has become a serious problem in public health; therefore, there is incentive to develop new bactericides. The development of nanotreatment strategies represents a opportunity to enhance medical treatments, improving prognosis and care for challenging healthcare issues [8]. Polymer matrix nanocomposites, which exhibit distinct physicochemical characteristics by incorporating inorganic fillers into polymer networks have received much attention due to their various industrial applications in drug delivery, water treatment, food industry, aeronautical and aerospace structures [9]. These nanocomposites combine advantageous properties of polymers with size-tunable optical, electronic, catalytic, and other properties of semiconductor nanoparticles. Generally, the role of the polymers is to encapsulate the nanoparticles and enable better exploitation of their characteristic properties [10,11]. A hybrid material consists of soluble polymers with inorganic component with excellent mechanical, optoelectronics and dielectric properties due to the combination of the organic and inorganic components. [12]. The

nanocomposites have different applications in many fields of medicine, antibacterial, thermal energy storage, optoelectronics, sensors, radiation shielding, piezoelectric, etc [13-25]. The effect of Silicon Carbide (SiC) nanoparticles on structural and electrical properties of (PVA-TiO₂) nanocomposites for antibacterial application has been investigated.

Materials and Methods

Films of polyvinyl alcohol (PVA)-titanium oxide (TiO₂)-Silicon Carbide (SiC) nanocomposites were prepared by using casting technique with concentrations: (98.5 wt.%) PVA and (1.5 wt.%) TiO₂ nanoparticles where dissolve 0.985 gm of PVA in 20 ml of distilled water by using magnetic stirrer to mix the polymers for 1 hour to obtain more homogeneous solution then 0.015 gm of TiO₂ nanoparticles was added to the PVA solution. The Silicon Carbide nanoparticles are added to (PVA-TiO₂) nanocomposites with different concentrations are (1.5, 3,4.5 and 6) wt.%. The electrical resistivity of (PVA-TiO₂-SiC) nanocomposites was measured at different temperature range from (40 to 80) °C using Keithly electrometer type,(2400source mater). The D.C electrical conductivity (σ) of,(PVA-TiO₂-SiC) nanocomposites calculated by following equation [26,27]:

$$\sigma = \frac{d}{R} \quad \dots\dots(1)$$

Where A = effective area,(cm²), R = resistance (Ohm),,d,= thickness of sample(cm).

The activation energy for,(PVA-TiO₂-SiC),nanocomposites is defined by[28,29]:

$$\sigma = \sigma_0 \exp \left(-E_{act} / K_B T. \right) \quad \dots\dots(2)$$

σ = electrical conductivity,at T temperature, σ_0 = electrical conductivity at absolute,zero of temperature, K_B = Boltzmann constant, E_{act} = activation energy,. Antibacterial activity of the nanocomposites tested samples was determined using a disc diffusion method. The antibacterial activities were done by using gram positive organisms (Staphylococcus aureus) and gram negative organisms (E. coli). Bacteria (S. aureus and E. coli) were cultured in Muller-Hinton Medium. The disks of the nanocomposites were placed over the media and incubated at 37 °C for 24 h. The inhibition zone diameter was measured.

Results and Discussion

The effect of Silicon Carbide (SiC) nanoparticles concentration on electrical conductivity of (PVA-TiO₂) is illustration in Fig.,1. The figure illustrates that the electrical conductivity is increased with increasing Silicon Carbide nanoparticles concentration, this conduct connected to the Silicon Carbide nanoparticles and,(PVA-TiO₂-SiC) nanocomposites having great number of free charge carriers obtainable for the aim of conduction. Hence, as the concentration of Silicon Carbide nanoparticles increases the number of free charge carriers also increases, as shown in Figs.2 and 3,all together electrons from polar O²⁻ terminated Silicon Carbide nanoparticles surfaces and from the,(PVA-TiO₂), follow-on the development in the conductivity of (PVA-TiO₂-SiC),nanocomposites,[30]. SEM images for (PVA-TiO₂SiC) nanocomposites for pure (PVA-TiO₂), (1.5 wt.%) and (6 wt.%), concentration of SiC nanoparticles to study the arrangement of nanoparticles and morphology of nanocomposites. SEM images demonstration that paths network formed of nanoparticles inside the (PVA-TiO₂),where charge carriers are allowable to permit by the paths.

Variation of electrical conductivity for,(PVA-TiO₂-SiC),nanocomposites with different temperature at altered concentration of Silicon Carbide nanoparticles demonstrations in Figure,4.,From the figure, the increase of conductivity for,(PVA-TiO₂-SiC),nanocomposites is exponential with temperatures. This is caused by thermal excitation of carriers into the conduction band. The electrical conductivity increases so as temperature shows many ions and electrons acquire kinetic energy via.,thermally activated hopping of charge carriers between trapped sites,, which is temperature dependence., It is suggested that in this region,, the band gap between conduction band and valence band is decrease meaningfully and provide easy for electrons to jumping from valence band to conduction band [31].The relationship between the ln(σ) and inverted absolute temperature of,(PVA-TiO₂-SiC),nanocomposites with altered concentrations of Silicon Carbide nanoparticles is shown in Fig.,5.

The activation energy of,(PVA-TiO₂-SiC),nanocomposites have high values for,(PVA-TiO₂),,this is connected to the presence of free ions in the polymer. Because of impact the space charge and forms local energy levels in the

forbidden energy[32],,the activation energy of D.C electrical conductivity for nanocomposites decreases with an increase the concentration of (SiC) nanoparticles,(as shown in, Fig.6).

In comparison with the results obtained theoretically by using Gaussian 09(G09) program and density functional theory (DFT) with (B3LYP/6-31G) basis set, the values of the total energies decrease with the increase in the number of atoms forming the nanocomposites. This result is fully compatible with a reducing values of effective energies, which were calculated in practice. When the number of atoms is (**46 Atoms**)(as shown in Fig.7),the value of calculated total energy (**-25821.776eV**), and by increasing the number of atoms to (**91 Atoms**) (as shown in Fig.8),the energy value is reduced to (**-53644.688eV**).So, the total energy values decrease with an increase number of atoms forming the composite.

The antibacterial properties of the (PVA-TiO₂-SiC),nanocomposites were examined against gram-positive,(Staphylococcus aureus),and gram-negative,(Escherichia coli) and the get data are show in Figs. 9 and 10.

There are some methods to make polymers antibacterial such as ionizing radiation, but they can be still infected by microorganisms during usage of them.The best and easy way to obtain antibacterial polymers is melt mixing of polymers with antibacterial agents[33].In this study, Nano TiO₂ and SiC particles were mixed with polymer (PVA).Antibacterial agents like TiO₂ has photocatalytic activity under UV, light so daylight or UV light is needed to make them active in killing bacteria[34].Many studies have been conducted in the field of photo catalysis where SiC filters were used as a structured photocatalytic backing[35].Numerous available works have shown that p-type SiC support meaning fully improves photo-catalytic properties owing to the obtain able heterojunction which seems when immobilizing an n-type semiconductor photocatalyst on it[36].when UV light was irradiated to the surface of the nanocomposites, the energy adsorbed exceeded the energy band gap of TiO₂ and SiC decrease, which made electrons transfer from the valence band to the conduction band and formed plenty of light electrons and holes on the surface of TiO₂ and SiC filter,[37]. The conduction band of SiC,(Si) is more negative than that of TiO₂, which makes the electrons created in the SiC conduction band transfer from SiC to

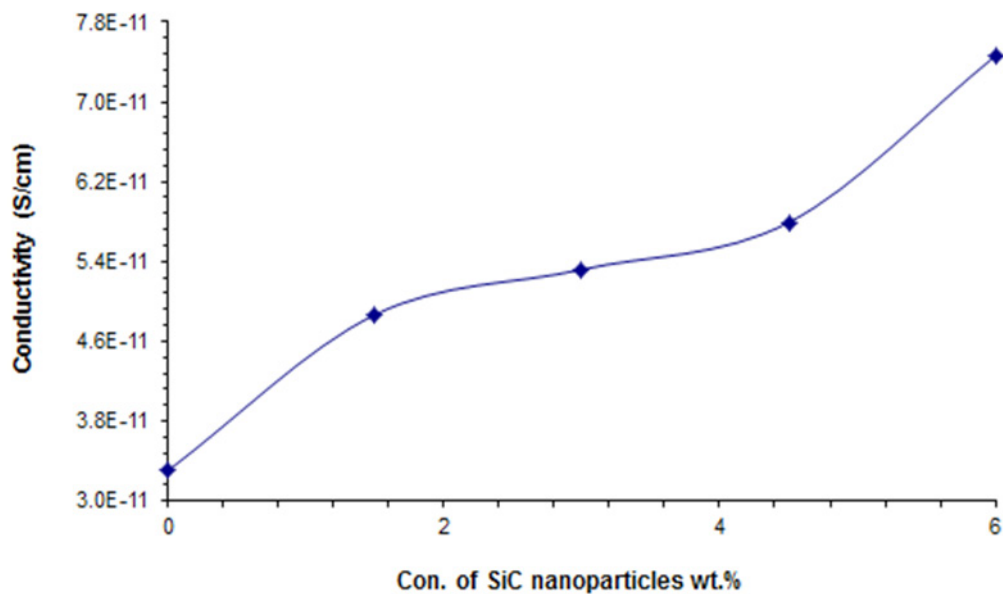


Fig. 1. Effect of SiC nanoparticles concentrations for (PVA-TiO₂-SiC) nanocomposites on D.C electrical conductivity.

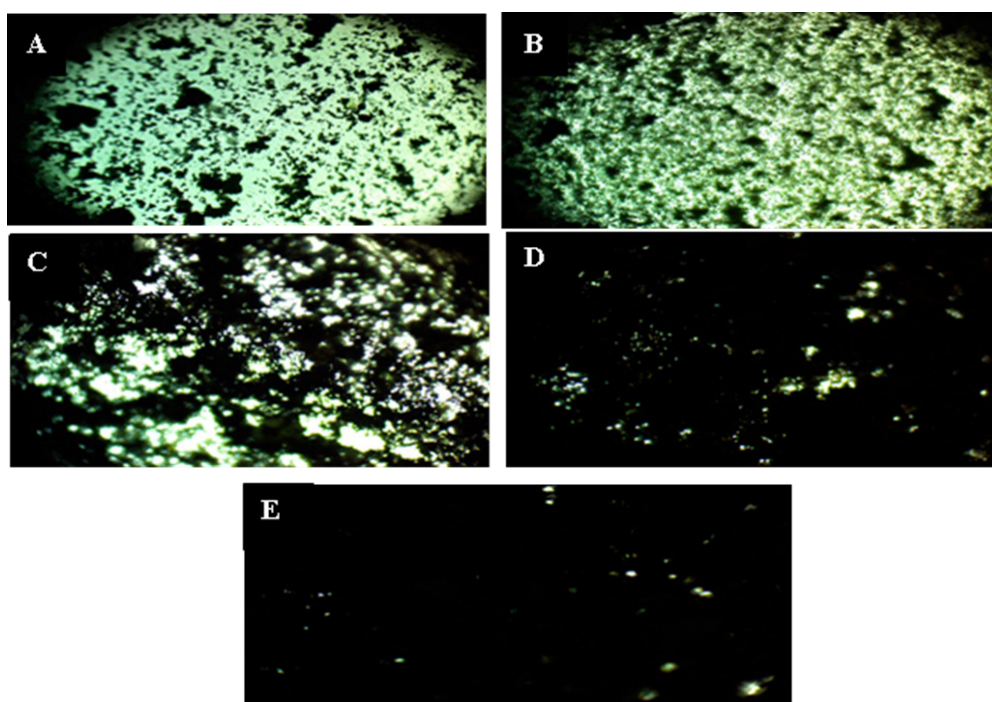


Fig. 2. Photomicrographs ($\times 10$) for (PVA-TiO₂-SiC) nanocomposites. A- For pure, B- for 1.5 wt% SiC nanoparticles, C- for 3 wt% SiC nanoparticles, D- for 4.5 wt% SiC nanoparticles, E- for 6 wt% SiC nanoparticles

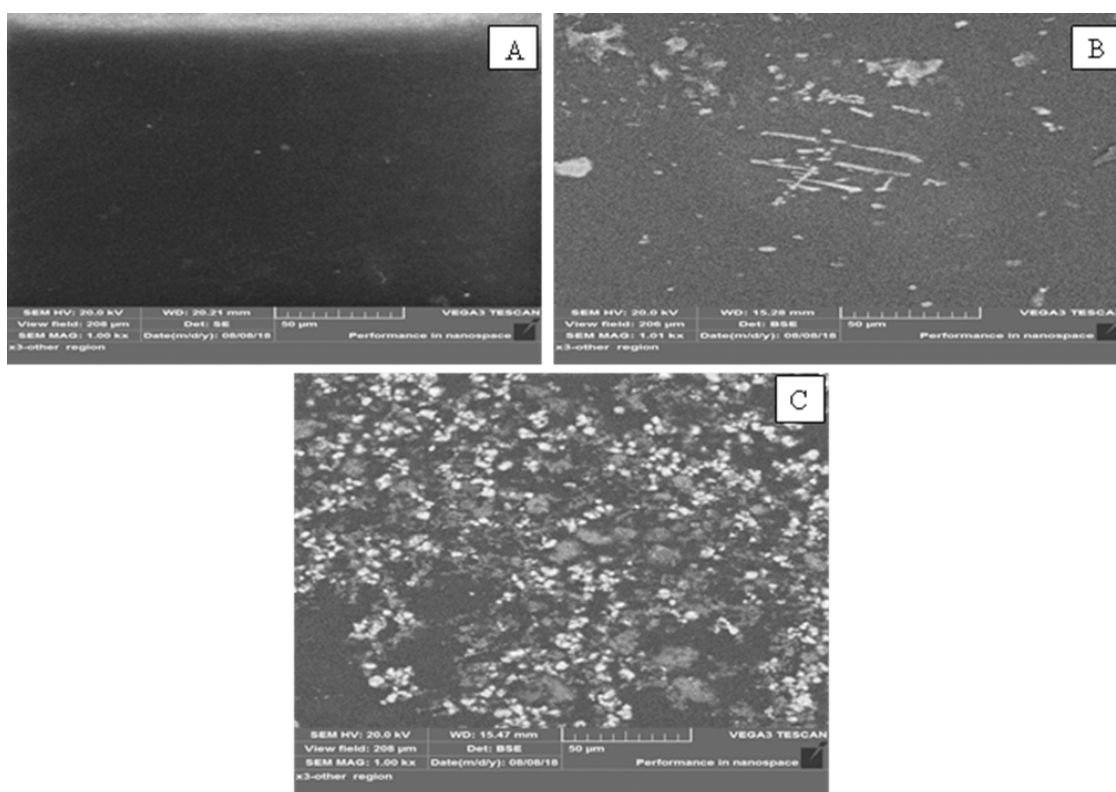


Fig. 3. SEM images for (PVA-TiO₂-SiC) nanocomposites : (A) for (PVA-TiO₂), (B) for 1.5 wt.% SiC nanoparticles; (C) for 6 wt.% SiC nanoparticles.

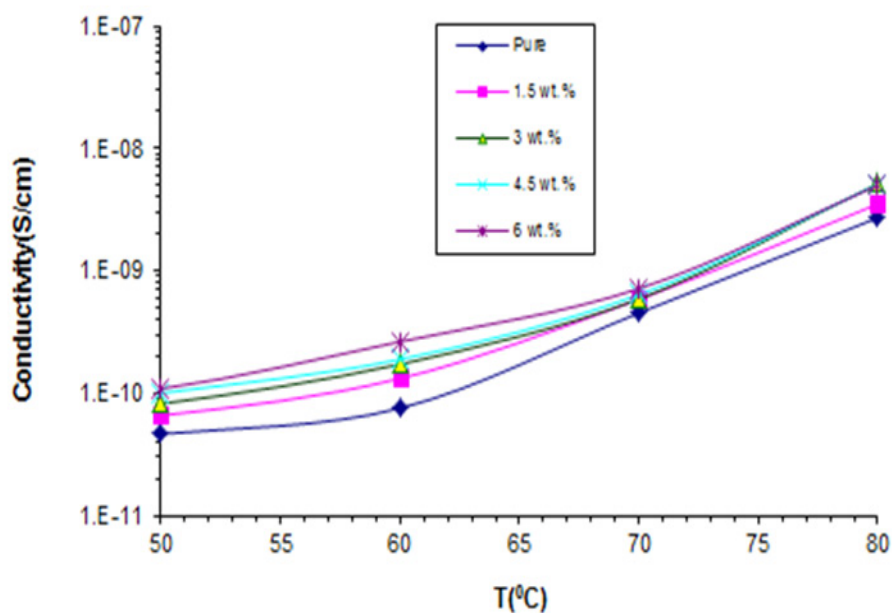


Fig. 4. Variation of D.C electrical conductivity for (PVA-TiO₂-SiC) nanocomposites with temperature

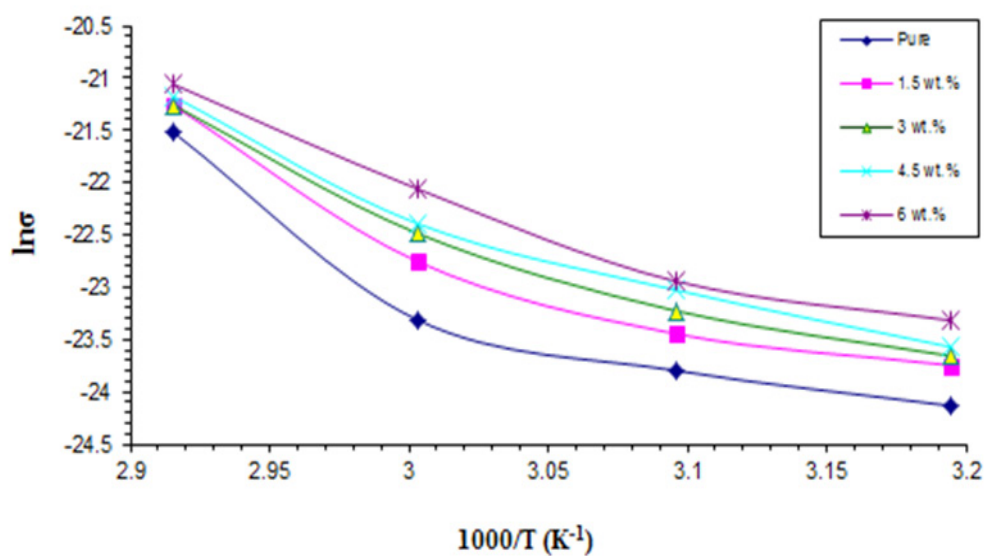


Fig. 5. Variation of $\ln(\sigma)$ and inverted absolute temperature for (PVA-TiO₂-SiC) nanocomposites

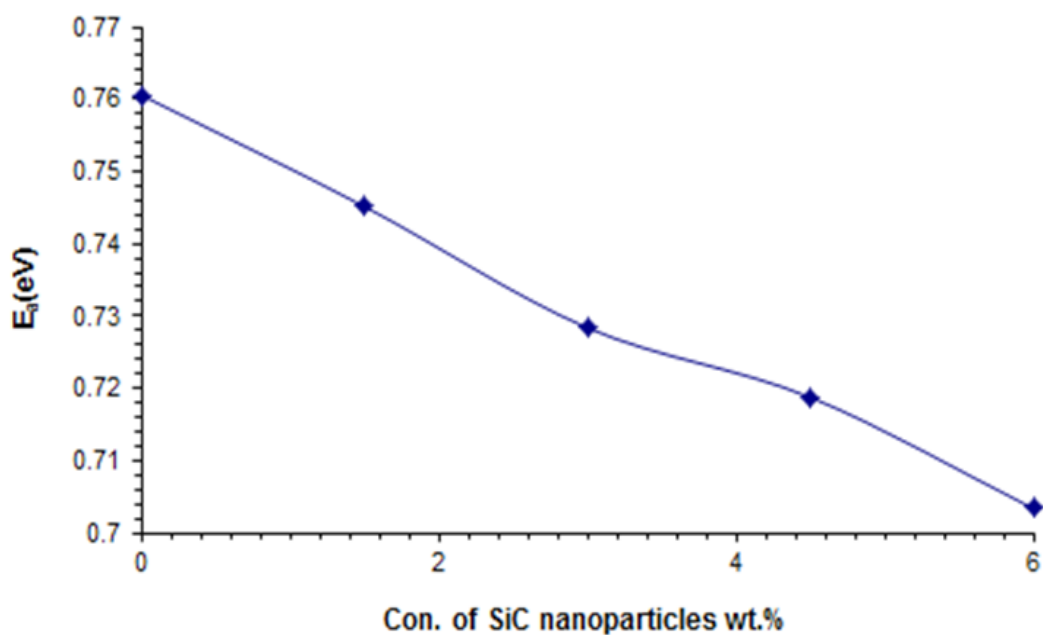


Fig. 6. Variation of activation energy for (PVA-TiO₂-SiC) nanocomposites with SiC nanoparticles concentrations

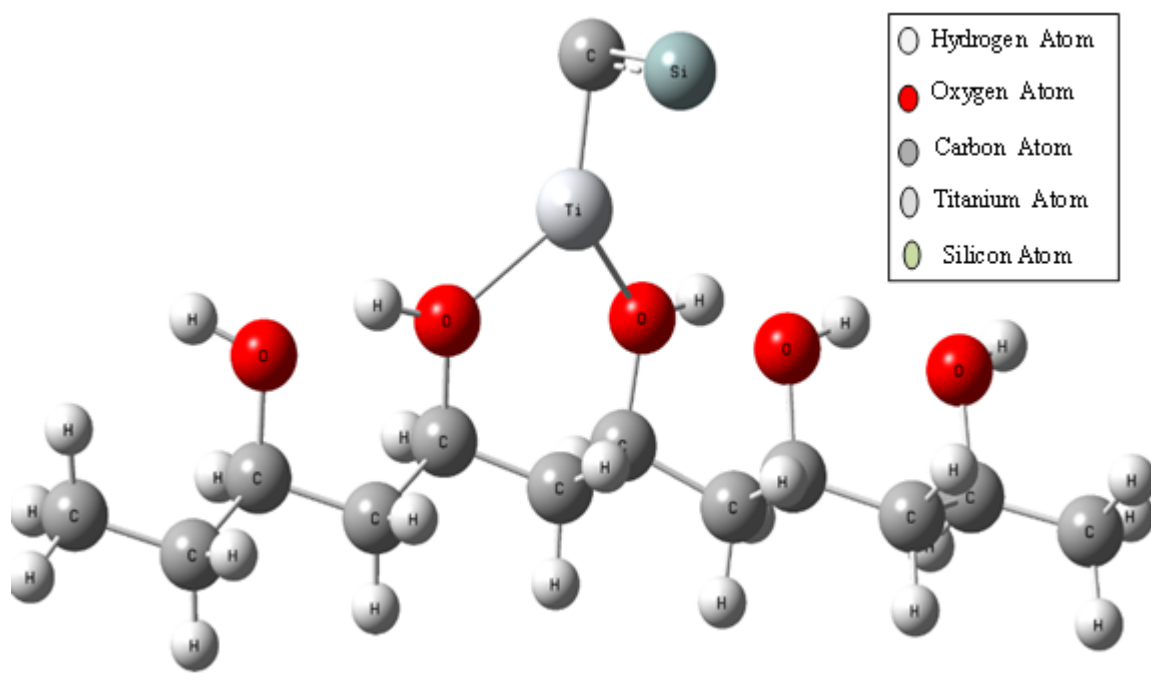


Fig. 7. Optimized geometry for (PVA-TiO₂-SiC) contain(46 Atoms) at the B3LYP/ 6-31G basis set

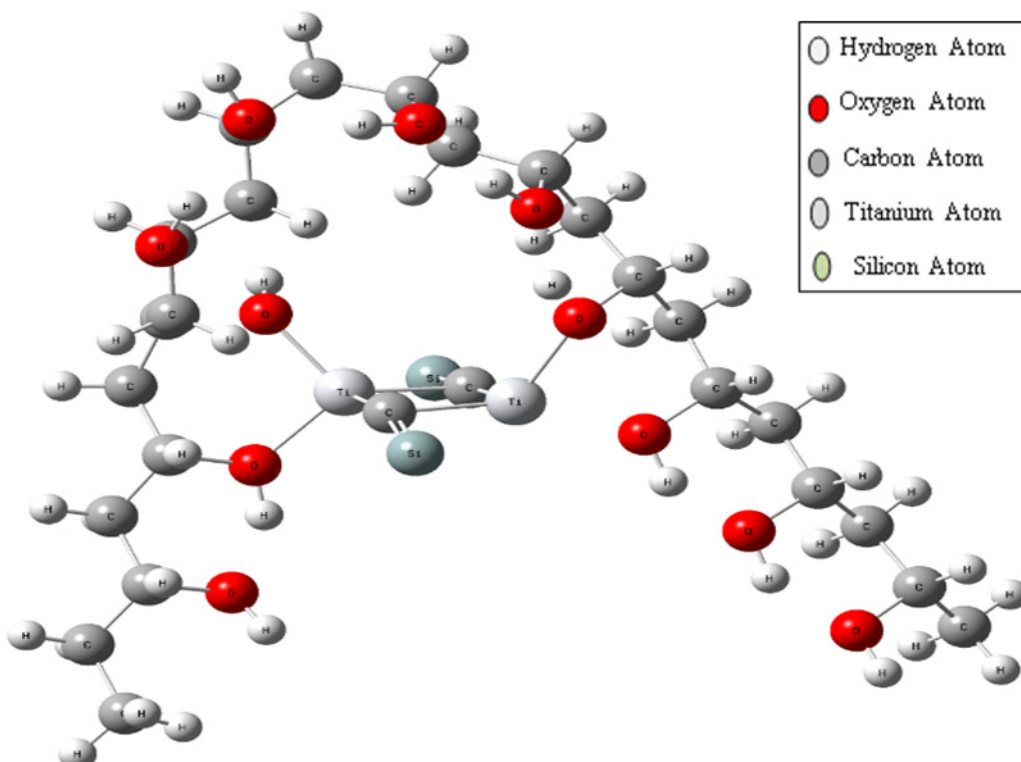


Fig. 8. Optimized geometry for (PVA-TiO₂-SiC) contain(91Atoms) at the B3LYP/ 6-31G basis set

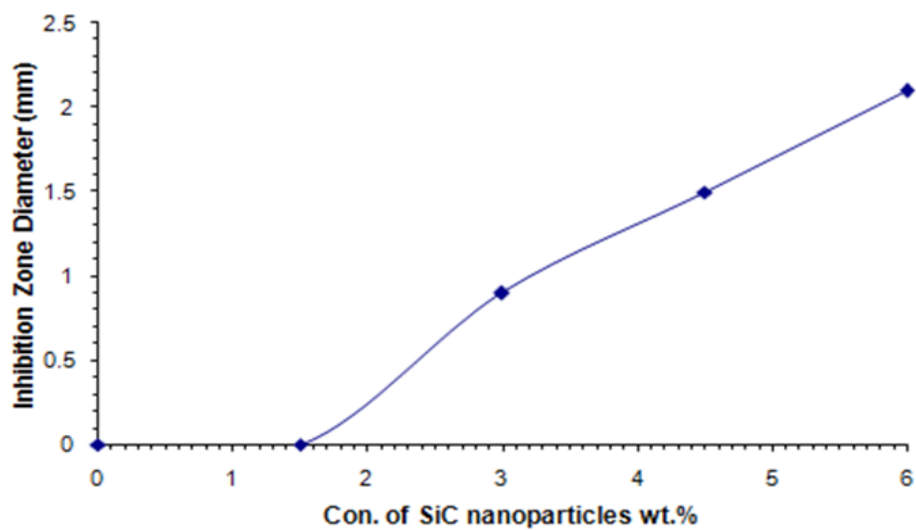


Fig. 9. Antibacterial effect of (PVA-TiO₂) blend as a function of SiC nanoparticles concentrations on *S. aureus*

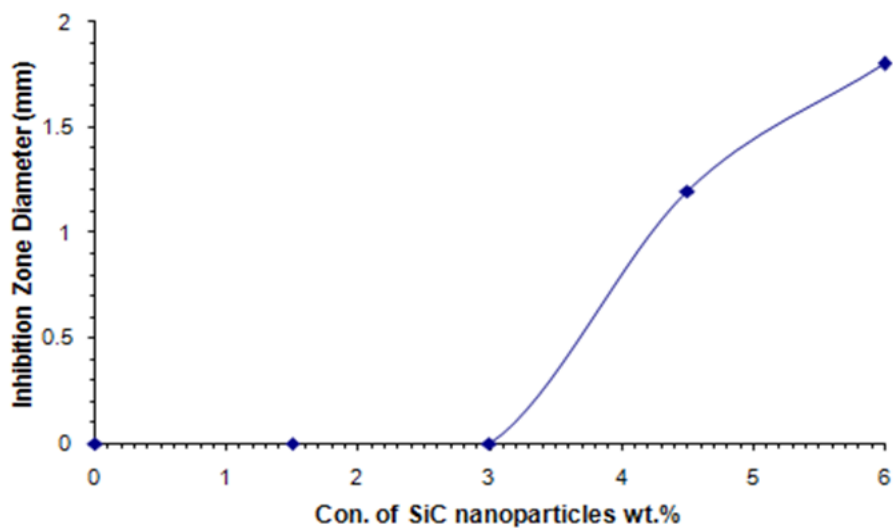


Fig.10. Antibacterial effect of (PVA-TiO₂) blend as a function of SiC nanoparticles concentrations on *E. coli*

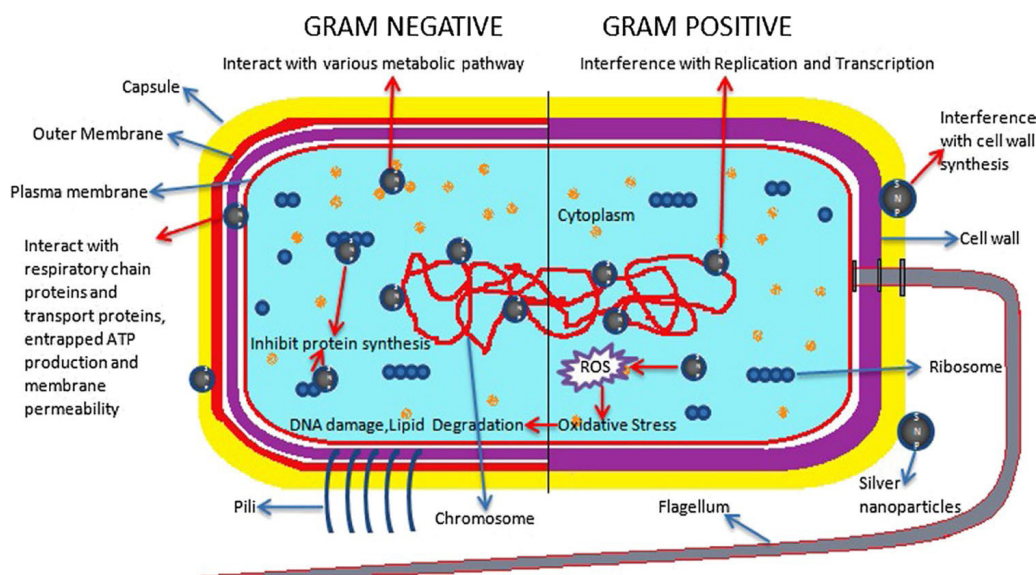


Fig. 11. Possible mechanisms of antibacterial activity of silver nanoparticles [39]

TiO₂ nanoparticles. The valence band of TiO₂ is more positive than that of SiC (Si), so the holes will transmit from the valence band of TiO₂ to that of SiC. So, a p-n junction is created in the interface of TiO₂ and SiC filter support. When the thickness of nanocomposites, coating surpassed an optimal value, UV light impossible permit through the nanoparticle layer, which Prevents the charge transmission and reduced the photo-catalytic performance. The valence band holes can oxidize great majority of organic pollutants into CO₂ and H₂O, and can interact with OH⁻ to generate, ·OH radicals, which additional oxidize numerous of the refractory organic pollutants into CO₂ and H₂O. The immobilization of TiO₂ nanoparticles on SiC filter efficiently enhances the photo-catalytic activity of supported TiO₂ photocatalysts and increases the feasibility of produce this method. The photo-catalytic degradation of organic contaminations and bacteria [36,38]. The TiO₂-SiC indicated beneficial photo catalytic performance as compared to TiO₂ nanoparticles, [36], therefore, the inhibition zone in Fig. 8 and 9 increases with an increase in SiC nanoparticles concentrations. The reason for the antibacterial activity of nanocomposites may be due to the presence of reactive oxygen species (ROS) generated by different nanoparticles. Chemical interaction between hydrogen peroxide and membrane proteins or between the chemical

produced in the presence of nanocomposites and the outer bilayer of bacteria could be the reason for the antibacterial activity of nanocomposites. The hydrogen peroxide produced enters the cell membrane of bacteria and kills them. The nanocomposites continue to be in interaction with dead bacteria once the hydrogen peroxide is generated; thus foiling further bacterial action and continue to produce and release hydrogen peroxide to the medium. The possible mechanism of action is that the nanoparticles in nanocomposites are in the negative charges which create the electromagnetic attraction between the nanoparticles and the microbes. When the attraction is made, the microbes get oxidized and die instantly. The main mechanism that caused the antibacterial of nanocomposites by the nanoparticles might be through oxidative stress caused by ROS. ROS includes radicals like superoxide radicals (O⁻²), hydroxyl radicals (·OH) and hydrogen peroxide (H₂O₂); and singlet oxygen (¹O₂) could be the reason damaging the proteins and DNA in the bacteria. ROS could have been produced by the present metal oxide leading to the inhibition of most pathogenic bacteria like *E. coli* and *S. aureus*. At low concentrations of nanoparticles, the interaction of particles with the cell wall of bacteria decreases and at the high concentrations of the particles, the aggregation probability of particles increases, as a result, the

effective surface to volume ratio of particles and so the resulting interaction between particles and the cell wall of bacteria decrease. It is clearly evident from the result that the antibacterial activity of the samples was notably stronger against Gram-positive *S. aureus* than Gram-negative *E. coli*. The stronger antibacterial activity against Gram-positive bacteria is due to the structural difference in cell wall composition of Gram-positive and Gram-negative bacteria. The Gram negative bacteria have a layer of lipopolysaccharides on the exterior, followed underneath by a layer of peptidoglycan. Furthermore, this structure helps bacteria to survive in environs where exterior materials exist that can damage them. On the other hand, the cell wall in Gram-positive bacteria is principally composed of a thick layer of peptidoglycan consisting of linear polysaccharide chains crosslinked by short peptides to form a three-dimensional rigid structure. The rigidity and extended cross-linking not only endow the cell walls with fewer anchoring sites for the nanoparticles but also make them difficult to penetrate [8]. Pandey et al. [39], studied the silver nanoparticles synthesized by pulsed laser ablation: as a potent antibacterial agent for human enteropathogenic gram-positive and gramnegative bacterial strains. They examined the antibacterial property of PLA-synthesized SNPs (as shown in Fig. 11) and suggested that it can be used as an effective growth inhibitor against various pathogenic bacterial strains in various medical devices and antibacterial control systems [39].

Conclusions

The D.C. electrical conductivity of (PVA-TiO₂),nanocomposites ,increased with an increase the SiC,nanoparticles concentration and temperature. The activation energy is decreased with an increase the concentration of the SiC nanoparticles. The results obtained theoretically by using Gaussian 09(G09) program and density functional theory (DFT) with,(B3LYP/6-31G),basis set showed that values of the total energies decrease with the increase in the number of atoms forming the nanocomposites,and this result is fully compatible with the low values of effective energies, which were calculated in practice. The inhibition zones for *S.aureus* and *E.coli* are increased with an increase the concentration of SiC nanoparticles. The results showed the PVA-TiO₂/SiC nanocomposites have good antibacterial activity which are make it used for antibacterial as bulk and coating materials.

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تصنيع وخصائص المتراكبات (PVA-TiO₂)_{0.5-x}/ SiC_x النانوية لتطبيقات الطب الاحيائي

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تم دراسة الخصائص التركيبية، والكهربائية والالكترونية للمتراكبات (PVA-TiO₂-SiC) النانوية لتطبيقها كمضاد للبكتريا مع قلة الكلفة، وخفة الوزن وفعاليتها العالية كمضادات للبكتريا. حضرت المتراكبات (PVA-TiO₂-SiC) النانوية بتراكيز مختلفة من المتراكبات (PVA-TiO₂) النانوية وجسيمات كاربيد السيليكون النانوية (SiC). طريقة الصب استخدمت لتحضير للمتراكبات (PVA-TiO₂-SiC) النانوية. جسيمات كاربيد السيليكون النانوية اضيفت الى متراكبات (PVA-TiO₂) النانوية ببتراكيز هي (١،٥، ٣، ٤،٥ و ٦) نسبة وزنية. درست الخصائص الكهربائية للمتراكبات (PVA-TiO₂-SiC) النانوية بدرجات حرارة مختلفة. بينت النتائج التجريبية ان التوصيلية للمتراكبات (PVA-TiO₂-SiC) النانوية تزداد مع زيادة تراكيز جسيمات كاربيد السيليكون النانوية (SiC). طاقة التنشيط للمتراكبات (PVA-TiO₂-SiC) النانوية تقل مع زيادة النسب الوزنية لجسيمات كاربيد السيليكون النانوية. درست الطاقات الكلية للمتراكبات (PVA-TiO₂-SiC) النانوية باستخدام برنامج Gaussian 09(G09) و النظرية الدالية للكثافة مع مجموعة الاساس (B3LYP/6-31G). الطاقات الكلية تقل مع زيادة عدد الذرات المكونة للمتراكبات النانوية. اختبرت المتراكبات (PVA-TiO₂-SiC) النانوية لتطبيقات مضادات البكتريا ضد بكتريا موجبة الغرام وسالبة الغرام. بينت النتائج ان المتراكبات النانوية المحضرة تمتلك فعالية جيدة كمضاد للبكتريا.