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Antimicrobials Nano-Fiber PVA Pure and PVA: TiO_2 for Filtration Applications

Suaad S. Shaker
Nanotechnology and Advanced Materials Research Centre, University of Technology/Baghdad .
Aus A.Najim
Nanotechnology and Advanced Materials Research Centre, University of Technology/Baghdad .
Email:aus772005ma@yahoo.com

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Abstract

In this study, polyvinyl alcohol composite polymer membrane was successfully synthesized pure and at different TiO_2 concentrations (1%, 3%). The diameter of the PVA–TiO_2fibers ranged from 100 to 200 nm. X-ray diffraction analysis indicated that the main crystal structure was amorphous and there are no any sign of Anatase or Rutile or Brookite when increased TiO_2 concentrations to3% .absorption properties of polymeric membranes doped absorb of increases compare with of the undoped membrane. PVA pure nano-fibers membrane less zone of inhibition than PVA : 0.1 TiO_2 nano-fibers membrane and PVA : 0.3 TiO_2 nano-fibers membrane against gram-negative(Pseudomonas and Salmonella) and gram-positive (Staphylococcus aureus (S. aureus)).

Keywords: nanofiber, polyvinyl alcohol, electrospining

البولي فينيل الكحول النقي والمشوب بثاني اوكسيد التيتانيوم ضد المايكروبات لتطبيقات الفلاتر

الخلاصة:

في هذه الدراسه ،تم تصنيع غشاء و بنجاح لبوليمر البولي فينيل الكحول النقيه والمشوبه بتراكيز مختلفه (1٪، 3٪) لاوكسيد التيتانيوم .قطر الفايبر لبولي فينول المشوب باوكسيد التيتانيوم يتراوح من 100 الى 200 نانو متر.اكدت نتائج حيود الاشعه السينيه ان معظم التراكيب البلوري كان عشوائي ولاتوجد اي اشاره على وجود اي طور من اطوار اوكسيد التيتانيوم (Anatase وRutile وBrookite) عندما ازداد تركيز الشوائب الى (3٪) . الخصائص البصريه للاغشيه البولمريه المشوبه اعلى امتصاص مقارنه مع الغير مشوبه .النانوفايبر النقي اقل منطقه تثبيط مقارنه المشوب (1٪، 3٪) باوكسيد التيتانيوم ضد نوعين من البكتريا السالبه (Pseudomonas and Salmonella) والموجبه (Staphylococcus aureus (S. aureus))

الكلمات المفتاحيه :نانو فايبر ،بولي فينيل الكحول ،الاكتروسبننك

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2412-0758/University of Technology-Iraq, Baghdad, Iraq

INTRODUCTION:

Nanomaterials have become a research priority as biotechnology; defense and semiconductor industries in particular, are interested in potential applications of nanotechnology. Substantial amount of research on nano-scale fibers, for instance, is being conducted due to their prospective application areas; such as tissue engineering, filters,microchips, micro air vehicles, and hydrophobic thin films

Electrospinning (also called electrostatic spinning) is one of the promising processes to produce nano-scale fibers from both synthetic and natural polymers. Electric forces are used to form fibers from material solutions or melts [1] .

Poly(vinyl alcohol) (PVA) has great application potential in biomedical field because it is a water-soluble, fiber-processable, and biodegradable polymer. Recently, electrospun polymer nanofibers have attracted a great deal of attention for applications in medical and healthcare fields [2].

Among the polymeric nanofiber, polyvinyl alcohol (PVA) has extensive application in air filtration, textile industry and paper coating due to its flexibility and good chemical and thermal stability [2].

TiO_2 was selected as our target because of its important role in environmental cleaning and protection due to its ability to photocatalyse and decompose harmful organic compounds and low cost[3].

TiO_2 usually used as powder in solution, which high photocatalytic efficiency however, the catalysts should be separated from the purified water after treatment. To solve this problem, aphotocatalytic membrane reactor has been developed using various membrane techniques [3]

For successful implementation of this ideal, metal-oxide nanoparticles must be well dispersed in the solvent and also compatible with the selected polymer to prevent phase separation.[4]

In this study, PVA nanofibers containing TiO_2 were electrospun from PVA solutions containing TiO_2. The PVA solutions were prepared by refluxing PVA/ TiO_2 aqueous solutions.

Experimental work:-

A-Materials.

PVA (99+% hydrolyzed, Mw=89,000–98,000) and TiO_2 (99.998%) were purchased from Fluka company.

i. Preparation of PVA/water Solutions Containing Ti Nanoparticles.

10 wt% aqueous PVA solutions with different amounts of TiO_2 were refluxed at various temperatures for 3 h in order to reduce the Ti+ ions into Ti nanoparticles. The weight percentage of TiO_2 in the solutions was calculated according to the weight of PVA.i

ii. Proceeding.

The PVA nanofibers containing TiO_2 were prepared by electrospinning the 10 wt% PVA/water solutions containing TiO_2.The electrospinning setup consisted of a syringe and a needle (ID=0.25 mm), an aluminum collecting plate, and a high-voltage supply (electrospining system , model :ESP200,Nano Nc).

The syringe pump connected to the syringe was used to control the flow rate. The PVA solutions were electrospun at a positive voltage of 16 kV, a working distance of 12 cm (the distance between the needle tip and the collecting plate), and a solution flow rate of 0.2 mL/h. This stationary collection procedure results in non-woven fiber mats as seen in Figure (1)

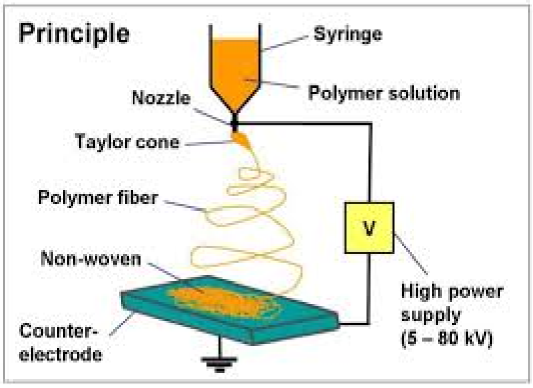

Figure (1) Electrospinning setup and its product: non-woven fiber mat.

B. Characterization techniques

The crystalline structure of the powder has been determined by using X-ray diffraction (Philips PW 1050 X-ray diffract meter of 1.5°A from Cu-Kα. Additionally. The surface morphology of nano fiber membrane was examined using Scanning Electron Microscopy (SEM ,the VEGA easy probe).The optical properties was examined using Optical absorption spectra of all the films were recorded using a UV-VIS spectrophotometer (Perkin Elemer Company).

C. Test for antibacterial activity of PVA pure and PVA : TiO_2

The antibacterial susceptibility of PVA pure and PVA doped with TiO_2 was evaluated using the zone inhibition method. A 100 μ L sample of bacterial suspension should be equal to McFarland solution, and using as a control that equal to 10^7 CFU/ml was then spread on a nutrient agar plate. The plates were then holed and supplemented with spherical of PVA pure and PVA doped with TiO_2 to determine the different antibacterial properties depending on sizes and shapes; the plates were then incubated further at 37 °C. The zones of inhibition were calculated after 24 h of incubation**.**

3-Result and Discussion

Figure (2) Show the XRD curve for various fibers samples. As shown in Figure 2a, Spectrum (a) of pure PVA shows an intense reflection peak at 2θ = 19.8˚ diffused in the hallow amorphous region and was assigned to a mixture of (101) and (10Í) reflections .The appearance of sharp reflections and diffuse scattering is characteristic of crystalline and amorphous phases of conventional semi-crystalline polymers . when doped with 0.01TiO_2 intense reflection peak shifted at 2θ = 19.6˚. It is observed that the increasing of TiO_2 to 0.03 concentrations lead to decreases intensity of PVA peaks and convert to amorphous. However, intensity of peaks became more flat with the increasing of the content of TiO_2 .The broader peak in the hybrid fibers indicates that the hybrid fibers are amorphous and there are no any sign of Anatase or Rutile or Brookite. These results revealed that the crystallinity of PVA was largely influenced by the content of (TiO_2) in the hybrid fibers [6] ,[7],[8].

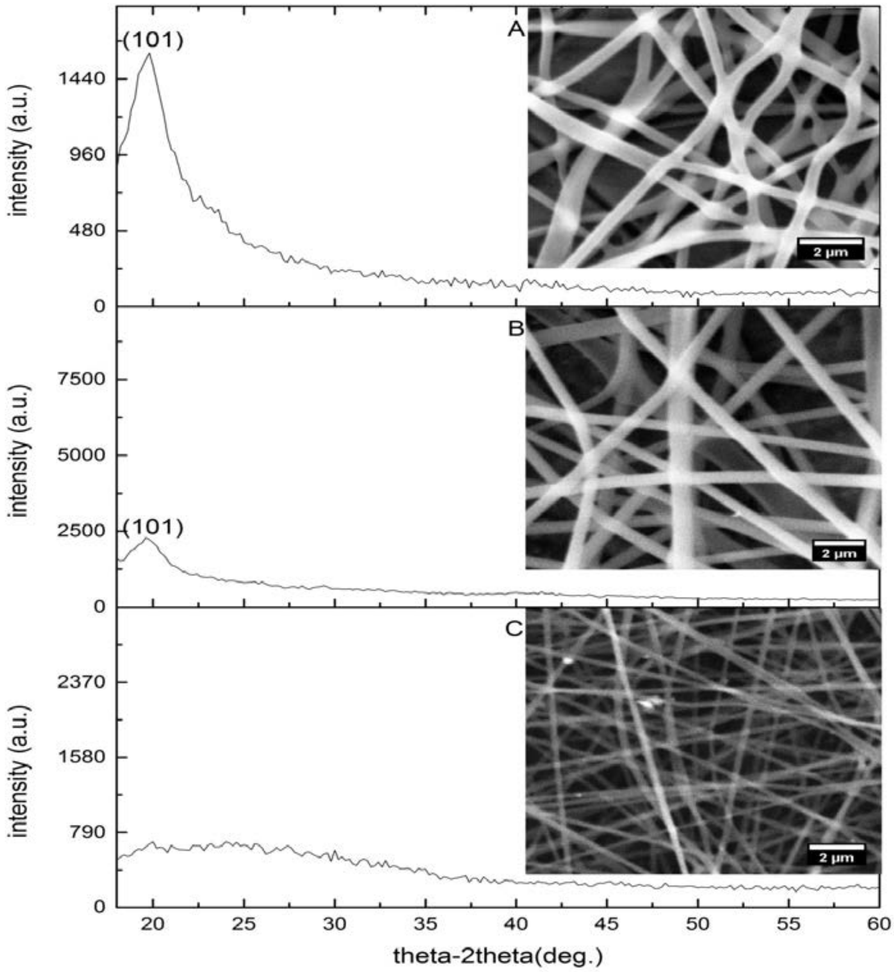

Figure (2) XRD curve for fibers samples with different mass ratio. .a) PVA pure b) PVA: 0.01TiO2 pure c) PVA: 0.03 TiO_2

The SEM images of hybrid fibers were displayed in Figure 3.(a,b and c) showed the smooth surface and uniform dimension of the fibers, with diameters of rang (100-200) nm .As a result, the PVA-3%TiO2 membrane was suitable for use in filtration application. From the SEM images (Figure 3) it appeared that the distance between cross fibers ranged from (1 - 5) micrometer. This was because the non-woven distribution of the fibers gradually decreased the size of the membrane pore. This kind of microstructure would be highly important for air and water purification processes since it can effectively entrap micro and nano-sized dust particles, such as viruses and bacteria. [5]

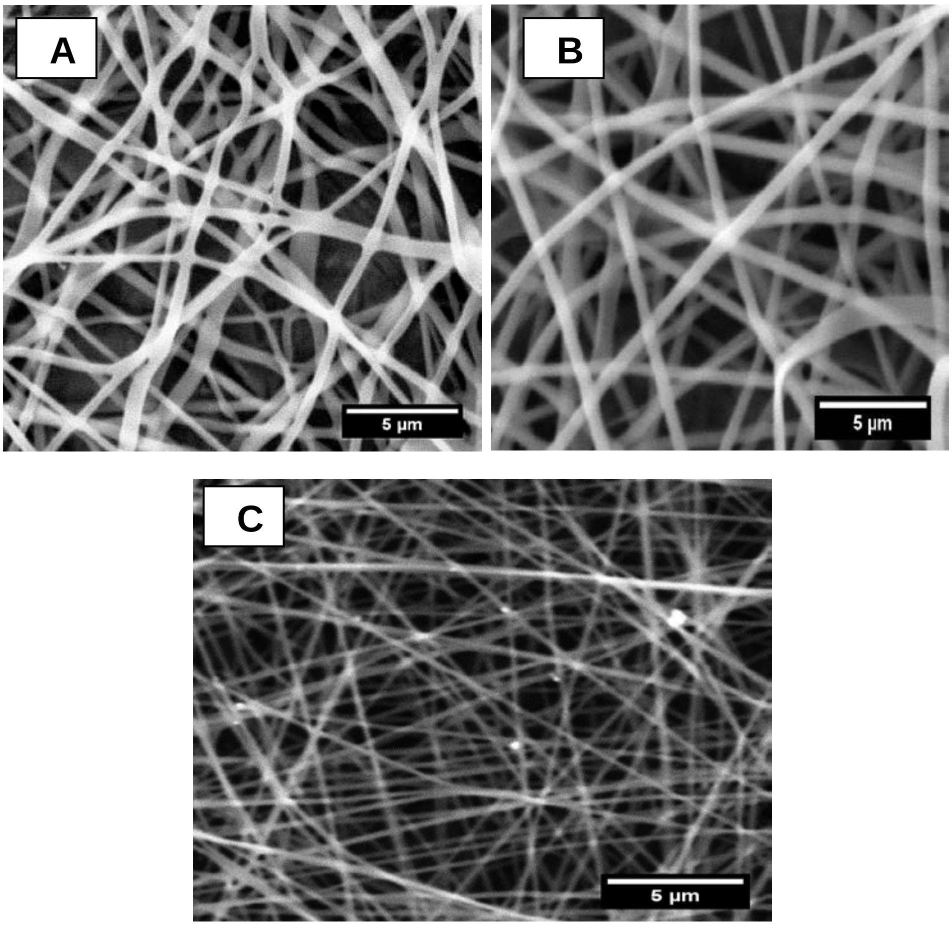

Figure (3) SEM images of (a) electrospun PVA nano-fibers membrane, (b) electrospun PVA-1%TiO_2 membrane, (c) electrospun PVA- 3%TiO_2 membrane,

The UV- Vis optical properties in the range from 300nm to 1100nm at various doping concentration, shows that the absorbance depends stronger on doping concentration as shown in Figure 4. At 1% concentration the absorption spectrum is very few modified. As the concentration of TiO_2 increases to 3% concentration, the absorption peak is amplitude increases compare with of the (PVA) undoped membrane, This behavior shows clearly the enhancement of absorption properties after addition of TiO_2.[9],[10].

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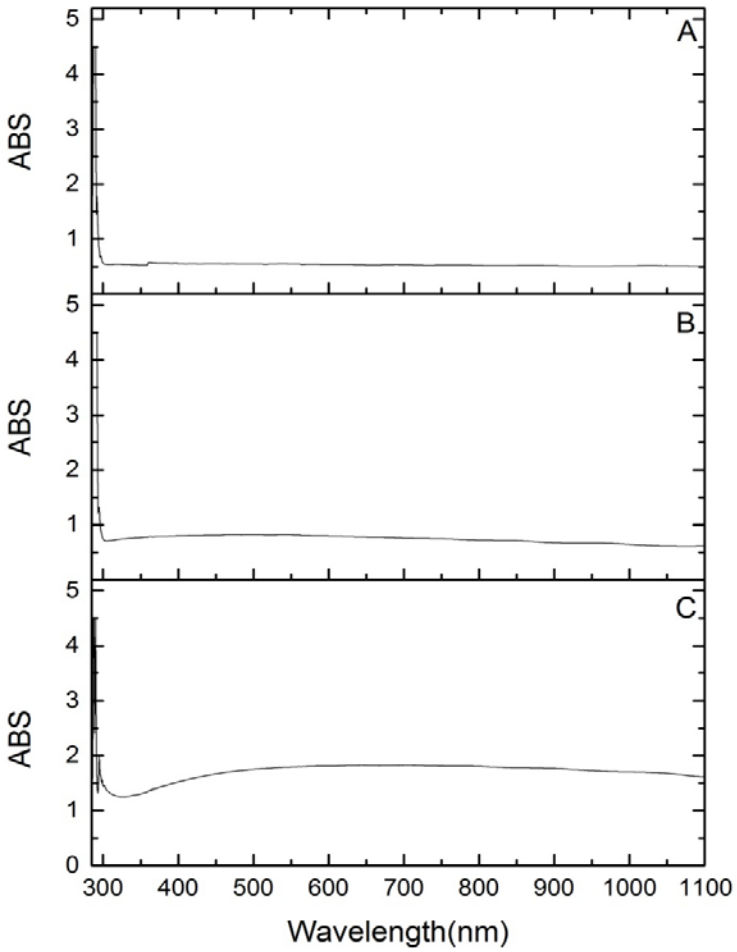

Figure (4) show absorption properties of polymeric membranes pure and doped with different concentrations of TiO_2.

Antibacterial checking by zone of inhibition (well diffusion assay) was carried out to qualitatively determine the level of inhibition using PVA nano-fibers membrane and PVA : 0.01 TiO_2 nano-fibers membrane and PVA : 0.03 TiO_2 nano-fibers membrane against gram-negative(Pseudomonas and Salmonella) and gram-positive (Staphylococcus aureus (S. aureus))bacterial systems Figure 6, shows the inhibitory effect PVA nano-fibers membrane and PVA doping with $TiO_2$and, can be observed as shown in Table 1 .

Table (1). The antimicrobial test results of treated PVA nano-fibers membrane and PVA : 0.01 TiO_2 nano-fibers membrane and PVA : 0.03 TiO_2 nano-fibers membrane .

BACTERIA / Material	Staph	Pseudomonas	Salmonella
PVA pure	5	8	8
PVA:0.01 TiO_2	14	16	16
PVA:0.03 TiO_2	15	17	18

From this table can be noticed PVA pure nano-fibers membrane less zone of inhibition than PVA: 0.01 TiO_2 nano-fibers membrane and PVA: 0.03 TiO_2 nano-fibers membrane against gram-negative(Pseudomonas and Salmonella) and gram-positive (Staphylococcus aureus (S. aureus))bacterial systems and PVA :(0.03, 0.01)TiO_2 membrane against gram-negative more zone of inhibition than gram-positive (Staphylococcus aureus (S. aureus)).

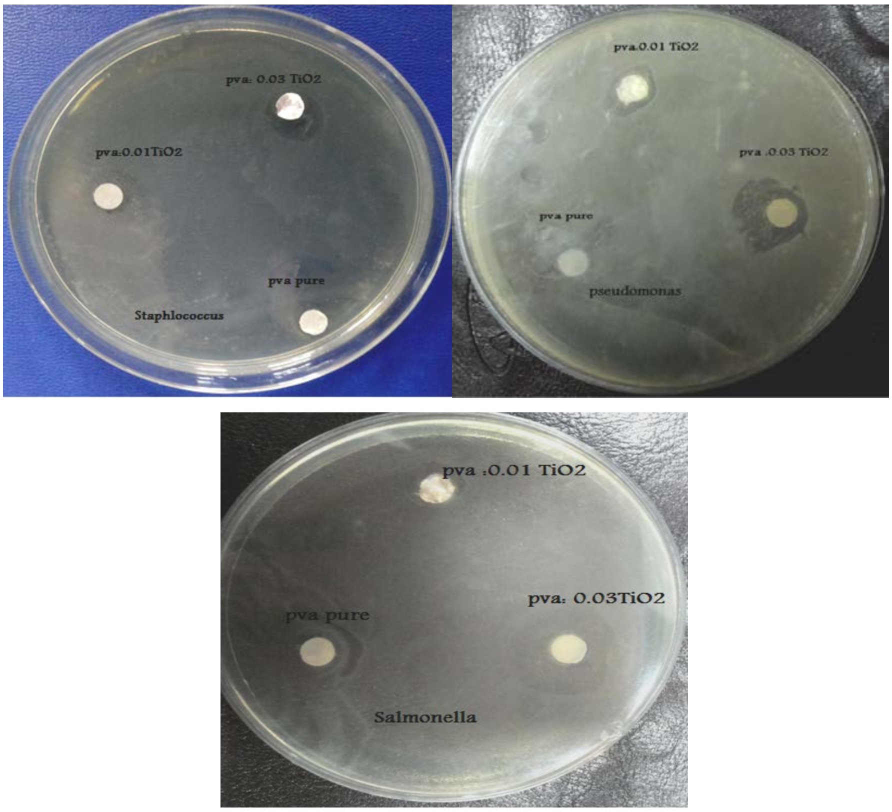

Figure (5) Antimicrobial activity of PVA nano-fibers membrane and PVA : 0.01 TiO_2 nano-fibers membrane and PVA : 0.03 TiO_2 nano-fibers membrane against Pseudomonas ,Salmonella and Staphylococcus aureus (S. aureus) shows the inhibition of growth after 24 hours of incubation.

Conclusion:

The surface morphology of of hybrid fibers have been studied by using scanning electron microscope showed the smooth surface and uniform dimension of the fibers, with diameters of rang hundred-two hundred nm and the pore size ranged one-five micrometer. UV-VIS absorption measurements have shown that the absorption peak is amplitude increases compare with of the (PVA) undoped membrane.

The study showed that PVA pure and doped fatal for both gram-negative and positive types of bacteria.

This kind of microstructure would be highly important for air and water purification processes since it can effectively entrap micro and nano-sized dust particles, such as

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