Preparation and Characterization of Cellulose Acetate /Ag-TiO2 Nanocomposite Films for Food Packaging Application

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Abstract

Nanocomposite film of Cellulose Acetate/Ag-TiO_2 were synthesized by cast method at different weight ratios of Ag nanoparticle (1.5, 2, 2.5) wt% and a constant weight ratio of 2 wt%TiO_2. The mechanical properties (Tensile strength and elongation) were improved at fixed level of CA+ 2%TiO_2 +1.5% Ag loading, beyond that level of loading, it decreased. The tensile strength was decreased due to some degrees of agglomeration of filler particles above a critical content. FTIR were conducted chemical composition of as-prepared composite films. The wettability of the films was also determined by Sessile drop method, an increase in contact angle was also observed by the addition of Ag content from 70.2° to 76.4° compared to pure CA that indicated a value of 61.3°. Antibacterial activity against Escherichia coli and Staphylococcus aureus were enhanced after incorporation of Ag-TiO_2 compared with pure CA. The enhanced wettability and antibacterial activity of the prepared films suggest that they could be used for Packaging applications.

Keywords: Antibacterial activity, biodegradable, biopolymer, Contact angle, Mechanical properties.

1. Introduction

Packaging technology provides endlenss opportunities for food preservation, and biopolymer-based films have been frequently employed in Packaging because of their edible, renewable, and biodegradable properties [1-5]. Due to benefits such as good appearance, biodegradability, excellent durability, and nontoxicity, cellulose acetate manufactured from naturally occurring cellulose materials such as wood, cotton, and rice husk is a green product nominated for producing food packaging films [6-8]. It's used in a variety of applications, including filters, drug delivery, and medical implants [9-12].Nanoparticles such as Ag, Au, Zn, ZnO, Titanum dioxide, and Cerium dioxide have been employed to make composite functional films to extend the functionality of packaging [13–15]. TiO_2 nanoparticles have gained a lot of interest because of their stable nature, ability to inhibt bacterial growth and prevent further formation of cell structures, environmental friendliness, low cost and good photocatalytic performance [16,17]. The US Food and Drug Administration (USFDA) has fully cleared the use of TiO_2 nanoparticles as food additives and food contact compounds after a safety assessment [18]. The incorporation of nanoparticles such as Au, Ag into TiO_2 nanostructures has been stated to improve visible light absorption due to high surface plasmon resonance excitation [19,20]. In comparison to Au nanoparticles and pure TiO_2 nanoparticles, SPR excitation can expedite the interfacial charge transfer between Au and TiO_2 nanoparticles, resulting in increased photocatalytic activity. [21–23]. Furthermore, Au-incorporated TiO_2 nanocomposites have been shown to have low toxicity [24]. Cellulose acetate films with Ag-TiO_2 nanocomposite integrated are made and applied to packaging in this study. The Ag-TiO_2 nanocomposite-incorporated films' physical and chemical properties, as well as antibacterial activity and contact angle, are all measured.

2. Material and methods

2.1 Materials

CA (CDH India, acetyl content 29-45%; Maximum limit of impurities; 0.1%; free acid (as acetic acid); 5.0%; loss on drying at 105℃ sulphated ash; 0.1%)) and Acetone AR/ACS (2-Propanone, Dimethylketone) (M.W.:58.08) from CDH India. Titanium oxide nanoparticles/nanopowder (TiO_2, Rutile, 99.5%), particle size (20-40) nm was purchased from Skyspring,USA. Hongwu international group ltd-china supplied Ag nanoparticle with particle sizes ranging from 40 to 80 nm.

2. 2 Methodology

2.2.1Preparation of primary polymer solutions

The CA primary solution was prepared by the dissolution of 7 g of CA in 100 ml of acetone using the casting method. The solution was then stirred for 8 h using a magnetic stirrer and then cast in a glass petri dish, and then a final film was obtained after 48 h.

2.2.2 Preparation of Ag/TiO_2/CA composite polymer solution

Ag and TiO_2 nanoparticles Solution were added to CA Powder at different weight ratio of Ag nanoparticle (1.5, 2, 2.5 wt%) and a constant weight ratio of 2%TiO_2 of composite film. The mixture was stirred until get the homogeneous solutions; Then the solution was casted in a glass Petri dish; and left to dry for 48 hrs. the preparation process was shown in Figure.1.

[insert Figure 1.]

Fig. 1. Schematic diagram of the preparation process for the films.

3. Characterization

The morphology of the powder was examined with FESEM Zeiss Sigma 300-HV Germany. The mechanical characteristics of the cellulose acetate and the composites thick films were measured by tensile tests using Laryee Universal Testing Machine (UTM) UE3450 from Laryee Technology Co., Ltd. China. The films were cut to 120 mm × 10 mm, and the thickness were measured by a digital micrometer with ±1 μm accuracy. All the tests were performed at room temperature (around 27^{o}C) and a crosshead speed of 5 mm/min. was used during the measurements of the cellulose based films, and a stress-strain curve was recorded. Young's modulus, elongation at break point and ultimate tensile strength were determined by a well-known standard procedure.

Fourier transformed infrared FTIR spectra were obtained by SHIMADZU-8400S FTIR spectrometer, Japan. The spectra were obtained in the wavenumber range of 400-4000 cm^{-1}, 4 cm^{-1} resolution. The samples were prepared as tablets by mixing with KBr powder.

The optical system used to measure contact angle was from Holmarc Opto-Mechatronics P Ltd. India, with automated dispenser and software for static and dynamic contact angle measurement. A drop of water is placed on the film surface and it will spread on the surface based on the interactions between the solid surface and the water. Water contact angle will be measured to give an indication of the wettability of the surface.

The antibacterial activity of the CA and CA/TiO_2/Ag was tested against human pathogens Escherichia coli and Staphylococcus aureus using the following protocol. The bacteria were captured from their stock cultures using a sterile wire loop. To assess how CA and CA/TiO_2/Ag have an impact on the bacteria's growth curve, the bacterial strains were grown at 37° C on M-H agar plates with immunization of 50 mL of nutrient broth. The bacterial growth grew up until the nutritional broth attained an optical density (OD) of 0.1 at 600 nm, which corresponds to a bacterial concentration of 108 CFU/mL. The bacterial cultures (1 mL) were then added to the nutritional broth along with CA and CA/TiO_2/Ag, and incubated at 37° C for 12 hours with slight agitation.The OD was measured with a spectrophotometer to determine bacterial growth. An unpaired t-test was used to statistically assess the obtained data. The results were provided as the mean ± SD of triplicate measurements.

Results and discussion

4.1 SEM analysis

SEM micrographs of TiO_2, Ag powders are shown in fig 2 a to b.

[insert Figure 2.]

Fig.2. SEM images of (a) TiO_2 nanoparticle, (b) Ag nanoparticle.

As clearly shown TiO_2, Ag nano powder have very fine particle size with narrow distribution of about 20-40nm and 40-80nm respectively. Fine particles have a high degree of agglomeration due to their high surface area, so a more sophisticated dispersion technique will be required to prevent agglomeration and to achieve a higher homogeneity of the particle distribution within the CA matrix.

4.2 Tensile test

The stress-strain curves for Ag-TiO_2 /CA composite films at different weight ratios of Ag nanoparticle (1.5, 2, 2.5) wt% and a constant weight ratio of 2 wt% TiO_2 are shown in Fig. 3. The good distribution of inorganic fillers in the (CA) polymer matrix was responsible for the improvement in mechanical properties by up to 1.5 wt% Ag and 2%TiO_2 [25-27]. The mechanical properties of polymer systems depend on the intermolecular force chain, stiffness, and molecular symmetry of polymer systems [28]. Polymers with a high degree of crystallinity, crosslinking, or rigid chains have a high strength or limited extendibility, resulting in a high yield modulus, a high stress at peak value, and a low elongation value. CA is a rigid, strong material that exhibits a dipole-dipole attraction due to electrostatic interactions between the ester atoms of one chain and the hydroxyl atoms of another. The interaction of dipole-dipole attraction, which reaches its maximum magnitude, was associated with the improvement in mechanical characteristics. The increase in strength can be attributed to an increase in the filler's capacity to adhere to the matrix, which resulted in less sliding between the composite layers when stress was applied to the composites [29,30].

[insert Figure 3.]

Fig.3. stress-strain curve of Ag/TiO_2 with Cellulose acetate.

The tensile strength is reduced due to some degrees of agglomeration of filler particles above the critical content and an increase in inhomogeneity [26, 27]. The lack of interfacial adhesion between the polymer and the fillers was responsible for the decrease in tensile strength [30].

At fixed level of 1.5%Ag- 2% TiO_2/CA loading; both tensile strength and elongation were improved as shown in Fig. 4, beyond that level of loading the values of tensile strength and elongation decreased.

[insert Figure 4.]

Fig.4. Ultimate tensile strength and Elongation of Cellulose acetate and TiO_2 films with different Ag content.

The general behaviour of Youngs modulus was found to be depended on the elongation and ultimate strength according to the filler contents and the homogeneity of particle distribution within the CA matrix.

[insert Figure 5.]

Fig.5. Young modulus of Cellulose acetate and TiO_2 films with different Ag content.

4.3 FTIR spectroscopy

Figures 6 show the FTIR spectra of pure CA film and CA films containing different weight ratios of Ag nanoparticle (1.5, 2, 2.5) wt% and a constant weight ratio of 2 wt%TiO_2. The film spectrum is characterized by the presence of bands at 1741 cm^{-1} (steric carbonyl stretching), 3478 cm^{-1} (cellulose OH stretching), and 2936 cm^{-1} (CH stretching) [31]. The bands of 2936 cm^{-1} (CH stretching), 3478 cm^{-1} (OH stretching), 1741 cm^{-1} (steric carbonyl stretching), 1232 cm^{-1}, and 1045 cm^{-1} (C-O stretching), all increased when Ag-TiO_2 nanoparticle were added. Because TiO_2 is an oxide with O-Ti-O bonding in its chemical structure, inserting it into cellulose acetate may have increased the interactions between the two compounds at the region of the band that represented these properties. TiO_2 bonding is represented by peaks at 600 cm^{-1} and 750 cm^{-1} [32,33]. Carbonian and hydroxyl groups have peaks cantered at 1750 cm^{-1} and 3478 cm^{-1}, respectively [32]. The vibration frequency of Ag–O ionic bond groups is indicated by the peak at 513 cm^{-1}. [34].

[insert Figure 6.]

Fig.6. FTIR spectra of Ag/TiO_2 based Cellulose acetate.

4.4 Water contact angle

The wettability test determines how many water molecules the sample absorbs. The wettability character of the surface is broadly defined as whether the sample is hydrophilic or hydrophobic. Water contact angles for CA and Ag-TiO_2 /CA at different weight ratios of Ag nanoparticle (1.5, 2, 2.5) wt% and a constant weight ratio of 2 wt%TiO_2 are represented in Fig. 7.

Because of its hydrophilic nature, the CA film had the lowest surface wettability of all the produced films, with a contact angle of 61.3°, as the low surface wettability leads to a high contact angle, and vice versa [35]. All composite films showed higher contact angles of 70.2, 72.4, and 76.4° compared to CA film. The contact angle of the prepared films increased towards hydrophobicity with an increase in nano Ag content in them [36].

[insert Figure 7.]

Fig.7. water contact angle for CA film and nanocomposite thick films with various Ag content.

4.5 Antibacterial activity

The optical density of Escherichia coli and Staphylococcus aureus suspensions at 600 nm treated with different films in test tubes for 12 hours was measured, which is a commonly used method for determining the growth of bacteria for the assessment of antibacterial activities [37,38]. In general, the lower the optical density of bacteria suspension at 600 nm after a specific period of cultivation, the better the antibacterial film's activity. As shown in Fig. 8, the OD600 values for various antibacterial films were in the following order: control> CA > CA/2%TiO_2/1.5%Ag > CA/2%TiO_2/2%Ag> CA/2%TiO_2/2.5%Ag . It's important to note that the nanocomposite film made up of CA+2% TiO_2+2.5% Ag had the lowest OD600. In contrast to other antibacterial films, this film performed particularly well after 12 hours of cultivation. These results clearly show that TiO_2 and Ag have a greater ability to resist bacteria growth [39-44].

[insert Figure 8.]

Fig.8. Effect of CA and CA/TiO_2/Ag in *E. coli* and *S. aureus* growth curve.

4. Conclusion

The present study used casting method to prepare Ag-TiO_2/ CA composite films. The tensile test proved that ultimate tensile strength and elongation were improved at fixed level of 1.5%Ag-2%TiO_2/CA, beyond that level of loading the values of tensile strength and elongation decreased. FTIR spectra demonstrated the interaction between CA and Ag-TiO_2. An increase towards hydrophobicity was also observed by the addition of Ag content. CA/TiO_2/Ag has enhanced antibacterial activity against Escherichia coli and Staphylococcus aureus compared with Pure CA. Due to the wettability properties and antibacterial activity of the prepared films, they could be used in packaging applications.

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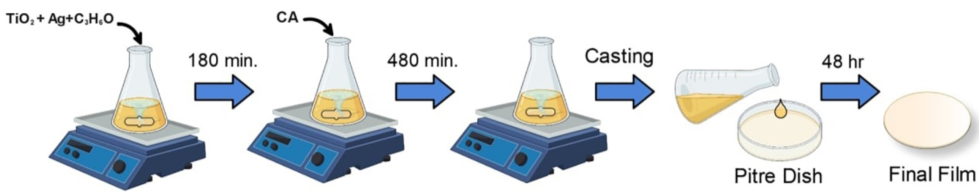

Fig. 1. Schematic diagram of the preparation process for the films.

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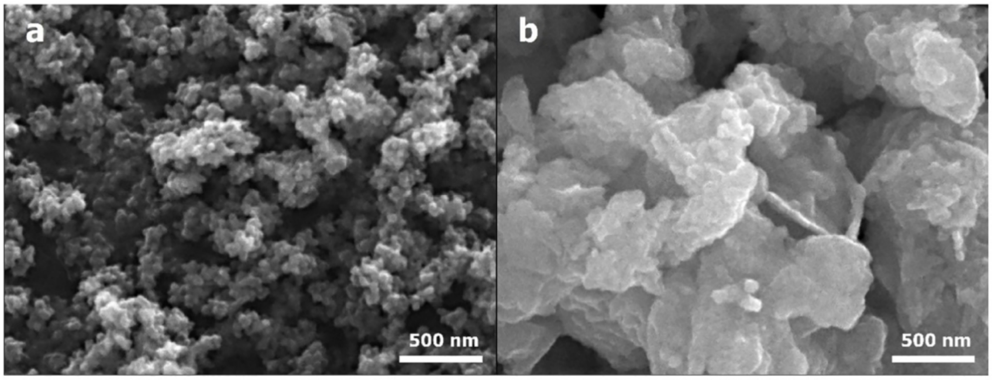

Fig.2. SEM images of (a) TiO2 nanoparticle, (b) Ag nanoparticle.

150x58mm (300 x 300 DPI)

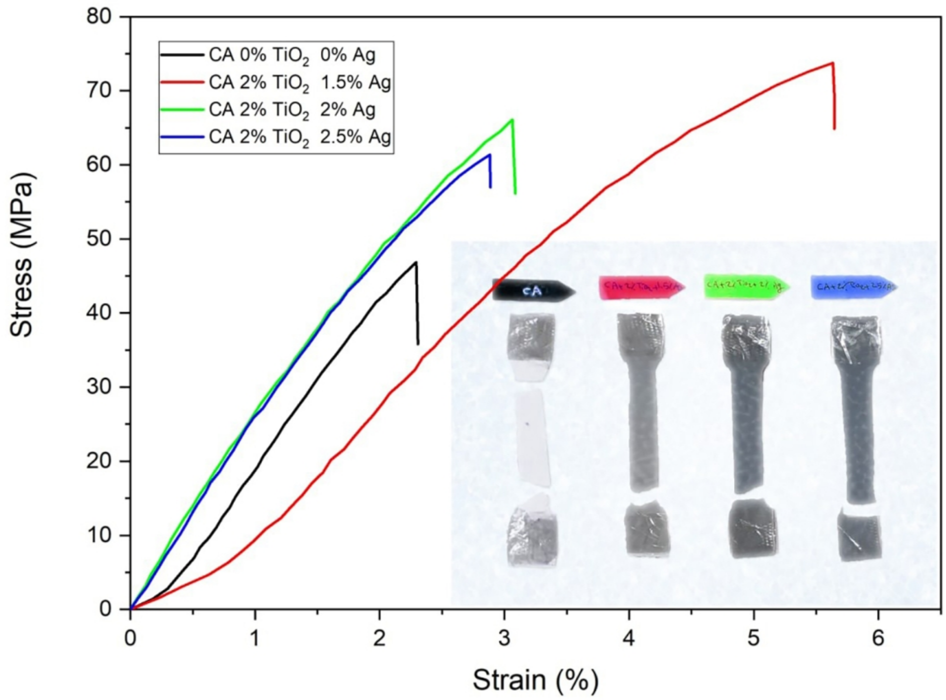

Fig.3. stress-strain curve of Ag/TiO2 with Cellulose acetate.

135x103mm (300 x 300 DPI)

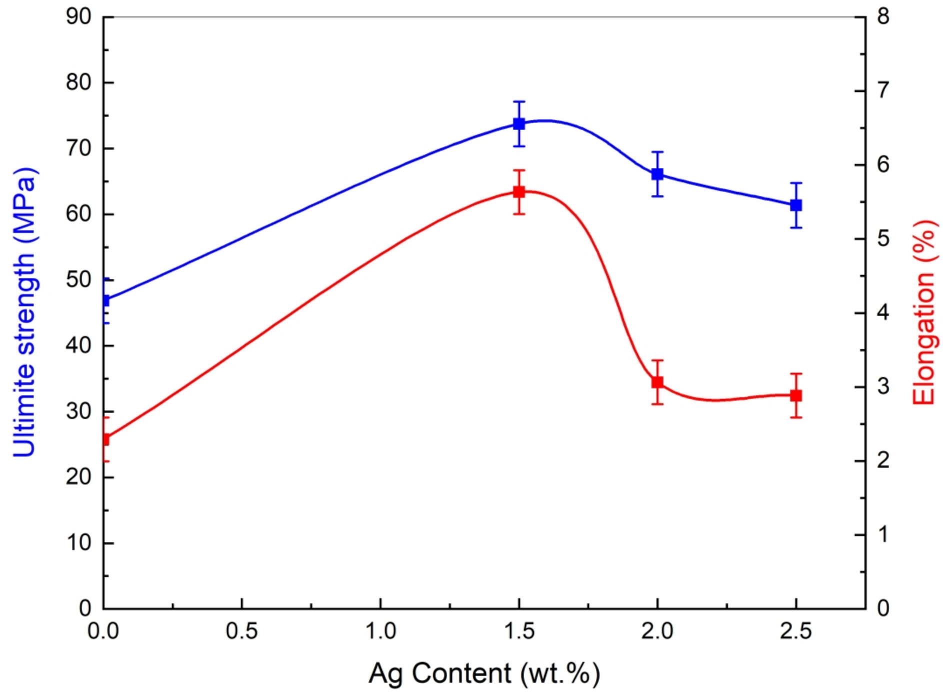

Fig.4. Ultimate tensile strength and Elongation of Cellulose acetate and TiO2 films with different Ag content.

272x208mm (300 x 300 DPI)

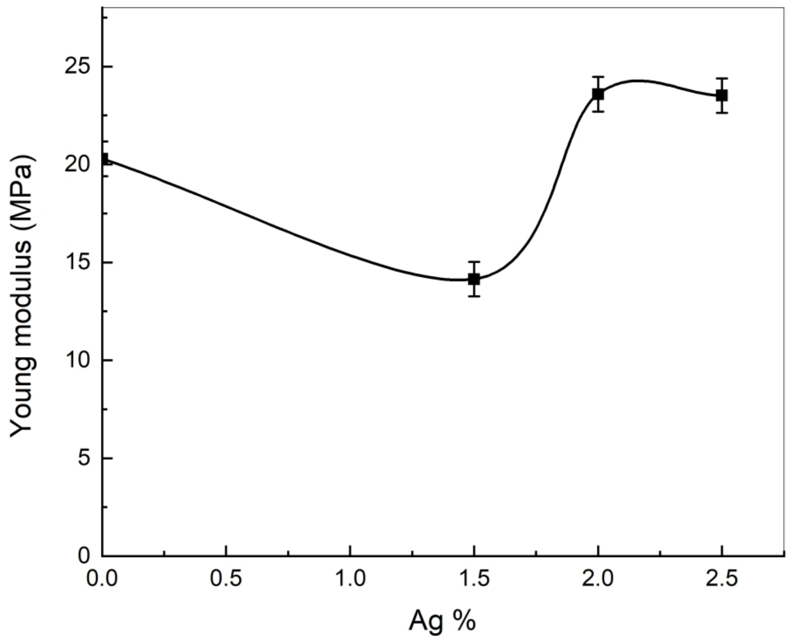

Fig.5. Young modulus of Cellulose acetate and TiO2 films with different Ag content.

272x208mm (300 x 300 DPI)

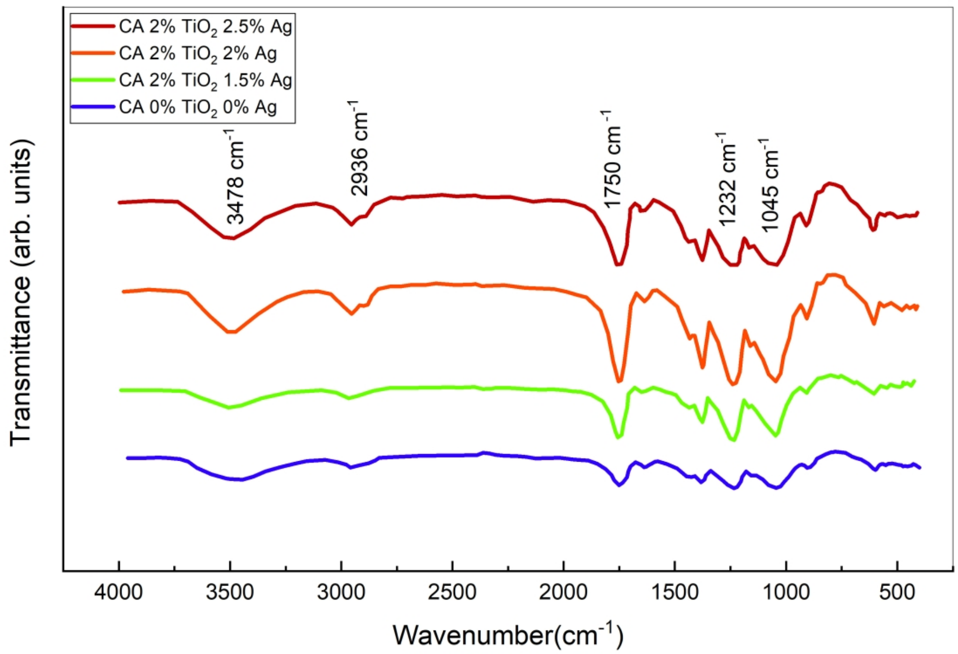

Fig.6. FTIR spectra of Ag/TiO2 based Cellulose acetate.

287x201mm (300 x 300 DPI)

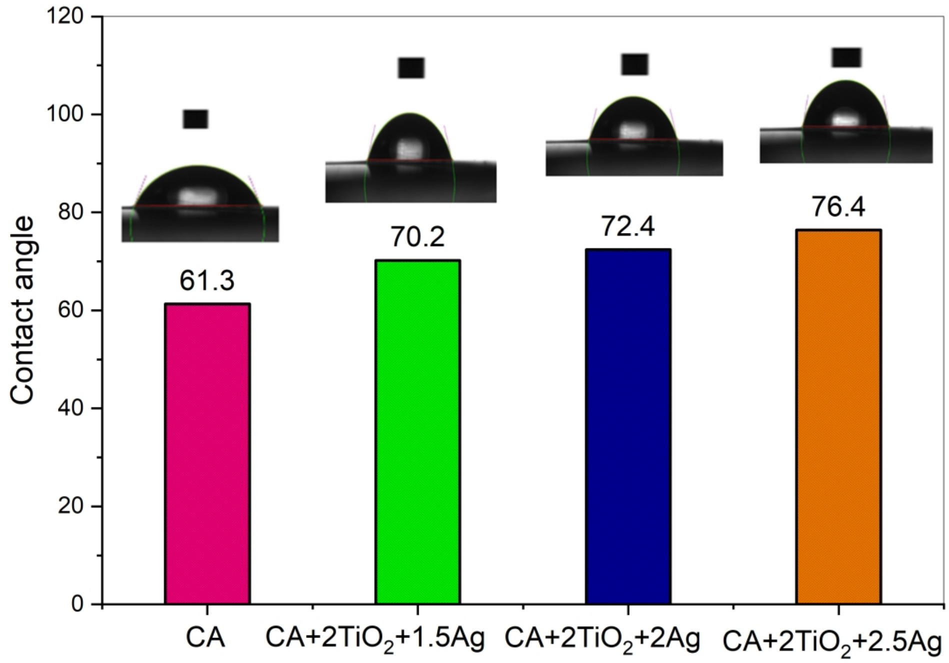

Fig.7. water contact angle for CA film and nanocomposite thick films with various Ag content.

272x208mm (300 x 300 DPI)

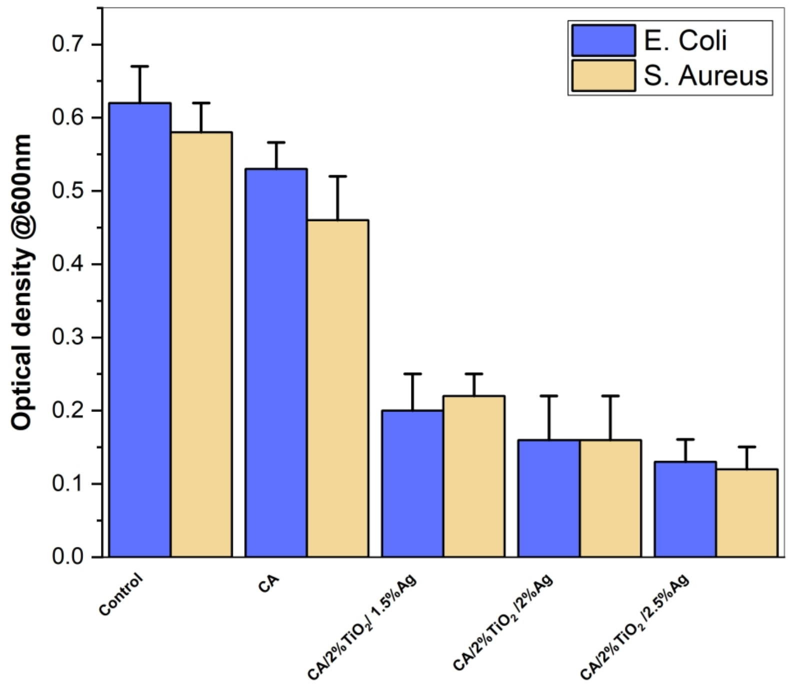

Fig.8. Effect of CA and CA/TiO2/Ag in E. coli and S. aureus growth curve.

272x208mm (300 x 300 DPI)