

Pulsed Laser Fabrication of FeS and FeS@Ag Core–Shell Nanoparticles for Antibacterial Applications

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Abstract: This study describes the preparation of FeS and FeS@Ag using a pulsed laser process with specific energy and frequency, followed by the deposition of the two materials using a special syringe onto porous silicon prepared by N-type photoelectron-etching. The prepared films underwent structural and optical characterization. X-ray diffraction (XRD) analysis confirmed the structure of both films, with the results indicating a crystalline size exceeding 10 nm. AFM and SEM analysis revealed aggregated iron spheroids coated with silver nanoparticles, forming tubes with dimensions exceeding 1 μm . These results confirmed that the prepared films were smooth and transparent with nanoparticles of varying sizes on their surface. High transmittance was observed across the surfaces of both films, increasing with wavelength and reaching a peak at 900 nm, which corresponds to relatively low absorption. The optical energy gap of the two membranes was calculated using FTIR spectroscopy to determine the effective FeS and FeS@Ag bonds. This was confirmed by UV-Vis and visible spectroscopy, which showed that both materials exhibited high absorbance in the UV range. These membranes were subsequently used as antibacterial agents. This study demonstrated the ease of preparing these materials and their effectiveness against a variety of serious pathogens in living organisms.

Keywords: FeS@Ag materials, X-ray diffraction, pulsed laser method, etching method of N-Type.

1 Introduction

Because thin films can combine physical and chemical properties in nanoscale dimensions, particularly in physics specialties, they are among the most prominent advanced materials in the domains of energy, electronics, healthcare, and others. Thus, a key foundation for nanotechnology is provided by the fusion of technology and materials science [1]. High-purity films have been produced thanks to contemporary manufacturing techniques including the sol-gel approach [2,3], the hydrothermal method [4,5], and the solid-phase method [6], particularly the pulsed laser method employed in this study [7,8]. As a result, very regular films have been produced and developed for a variety of functional uses, including optical, electrical, and medicinal [9]. applications in the fields of electronics, optics, and medicine [10]. The usage of nanomaterials with antibacterial qualities has drawn more attention in recent years as a way to address one of the major issues facing world health: bacterial resistance to antibiotics. Due to their great capacity to break down bacterial cell walls and interfere with essential functions within them, silver particles and films are among the most well-known of these materials [11]. Because of their catalytic activity, metal sulfide compounds like iron sulfide have also demonstrated encouraging potential. The

discharge of metal ions that might damage dangerous cells through oxidative stress. A novel technique for creating extremely effective antibacterial surfaces is the application of thin layers of silver and iron sulfide using a pulsed laser. It is anticipated to help improve these two materials' physical and chemical characteristics in order to increase the films' efficacy against a variety of dangerous bacteria while preserving their structural stability and the variety of their industrial and medicinal uses [12]. According to recent research, the antibacterial capabilities of silver are significantly enhanced when combined with iron sulfide. For instance, scientists created silver-modified hybrid nano-membranes that demonstrated superior chemical stability and strong bacterial growth inhibition [13]. Therefore, the creation of hybrid thin films of silver and iron sulfide using the pulsed laser method is a promising scientific approach to creating multifunctional materials that combine structural stability and antibacterial efficacy, opening up new possibilities for biomedical applications and surface treatment to fight infection. Because of its capacity to create high-purity films with regular crystal structures [14] that are appropriate for medical applications [15,16], it is thus confirmed that the pulsed laser method is not only a technique for material deposition but also a strategic tool for controlling the crystal structure and functional properties of

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thin films. Alkali telluride films have also been successfully prepared using this method to provide high-efficiency cathodes for ultra-bright electron sources [17]. In order to assess the activity of these materials against common bacterial strains like *Escherichia coli* and *Staphylococcus aureus*, the goal of this research is to successfully combine the antibacterial qualities of silver with the photocatalytic properties of iron sulfide prepared by pulsed laser method.

2 Experimental

The first step in the practical portion involved using pulsed laser technology to prepare iron sulfide nanoparticles (99.9% purity, APS: 30-45 nm pure composition, Nano shell, India, 1:1 molar ratio of Fe to S). This involved grinding 8 g of the material into an 8 mm diameter iron mold and pressing it under 5 tons of pressure for 30 minutes. Following completion, the material was placed in a glass container with five tons of microliters of deionized water, and an Nd: A YAG laser with a wavelength of 1064 nm, and an ablation energy of 600 mJ, and 600 pulse counts at an 8 Hz frequency was utilized. Following that, we arrived at the last phase of this process, which is to produce an iron sulfide nanofluid that is yellowish, as seen in figure (1). The iron sulfide substance was then coated with a silver alloy that weighed 3 by 10, had a thickness of 2, and had the same number of strokes. Using the photoelectrochemical etching approach with a current density of 15 ml/cm² and an etching period of 15 minutes, porous n-type silicon was prepared in the third step. In the last step, both FeS and FeS@Ag were deposited on porous silicon using a customized syringe and the drop casting technique. The samples were prepared for testing after five drops of each substance were applied to the porous silicon and allowed to dry for fifteen minutes.

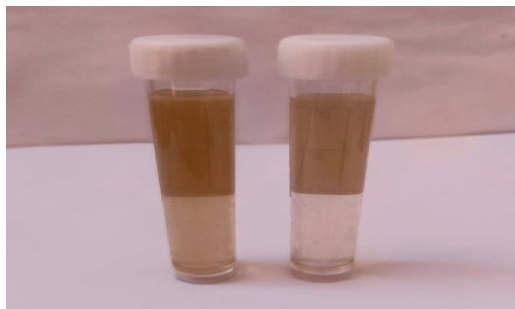


Fig. 1: the material from the left in order are FeS and FeS@Ag

3 Results and discussion

3.1 Structural Analysis

The structural properties (X-ray diffraction) examination in Figure (2)(a),(b) shows the crystal structure of the two materials prepared using pulsed laser technology and the drop-casting method, which has proven successful and suitable for preparing the two materials in this research. The figure shows the X-ray diffraction of the hexagonal shape of

the material (FeS, FeS@Ag) [18,19], with peaks appearing at angles $2\theta = (29.6, 23.7, 20.9)$ with diffraction coefficients (100,111,001) respectively, and matching JCDPS card No. (3356-065-03). The X-ray diffraction at angles $2\theta = (31.7, 29.8)$ are consistent with the X-ray diffraction coefficients (111,102) and matching X-ray diffraction card No. (1404-06-03). The crystal size, microstructural flexibility, and dislocation density were also calculated for both materials (FeS, FeS@Ag) by using the following equations Debye-Scherrer's equations $G.S = 0.9\lambda / \beta \cos$ [20,21] and $\delta = 1/GS^2$, $\eta = \beta \cos\theta/4$ equations [22] as it shown in table(1)and(2).

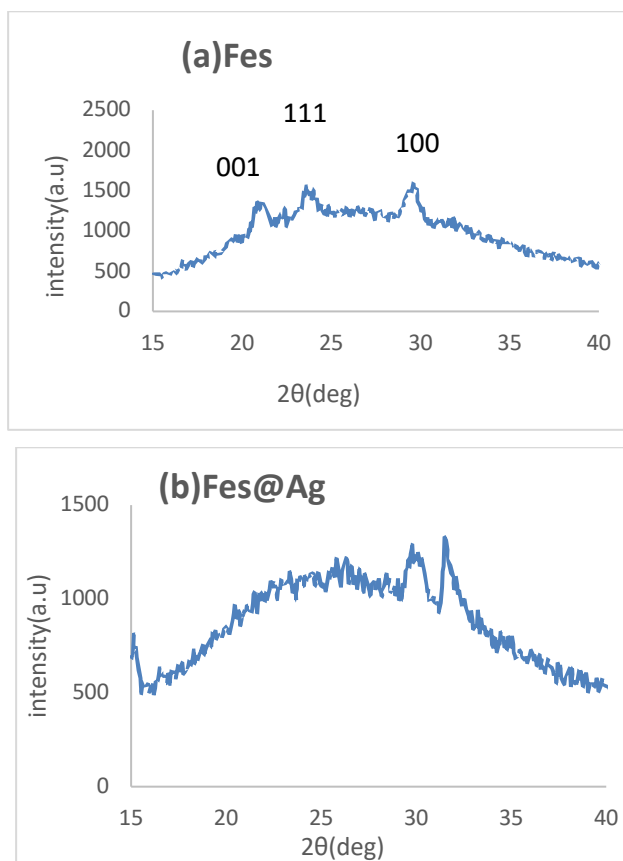


Fig. 2: XRD (a) of the FeS and(b) of the FeS@Ag

Table 1: XRD parameters of FeS

Pos. [°2Th.]	(hkl)	G.S (nm)	$\eta * 10^{-2}$	$\delta * 10^{15}$ (lines/m ²)
20.9	001	11.1	3.24	8.03
23.7	111	1.83	19.6	295.6
29.6	100	10.7	3.37	8.69

Table 2: XRD parameters of the FeS@Ag

Pos. [°2Th.]	(hkl)	G.S (nm)	$\eta * 10^{-2}$	$\delta * 10^{15}$ (lines/m ²)
29.8	102	9.40	3.84	11.31
31.7	111	2.68	13.47	138.5

3.2 FESEM Morphology

The scanning electron micrograph of the iron sulfide film

produced using pulsed laser technology is shown in Figure 3(a). The analysis showed that nanosheets, about 20 μm in size and 4.2 mm in breadth, appeared as clusters of other particles of various sizes, evenly distributed. This is explained by the film's quality, surface topography, and the benefits of using pulsed laser technology to prepare these films, which increase their surface area and roughness [23, 24]. Regarding the fes@ag membrane, figure (3) (b) demonstrates the enhanced surface characteristics because the silver nanostructures gave the membrane strong sensors to identify contaminants and dangerous bacteria, as silver ions are recognised to have antimicrobial qualities. With the appearance of particles ranging in size from 0.1 μm to 1 μm

and a width of 4.2 mm, the membrane exhibits greater stability due to steric interactions and its hybrid surface. This indicates that the silver particles are uniformly distributed on the membrane's surface. Fes@Ag is composed of Fes and Ag nanoparticles [26][27]. This contrasts with the findings of other studies, as the surfaces of these films exhibited distinct behaviour, with extreme roughness and comparatively low permeability, which altered the manufactured films' specific absorbance. This difference may be attributed to the thickness of the silver used in this research, which led to the formation of films with finer particles distributed on the surface [25].

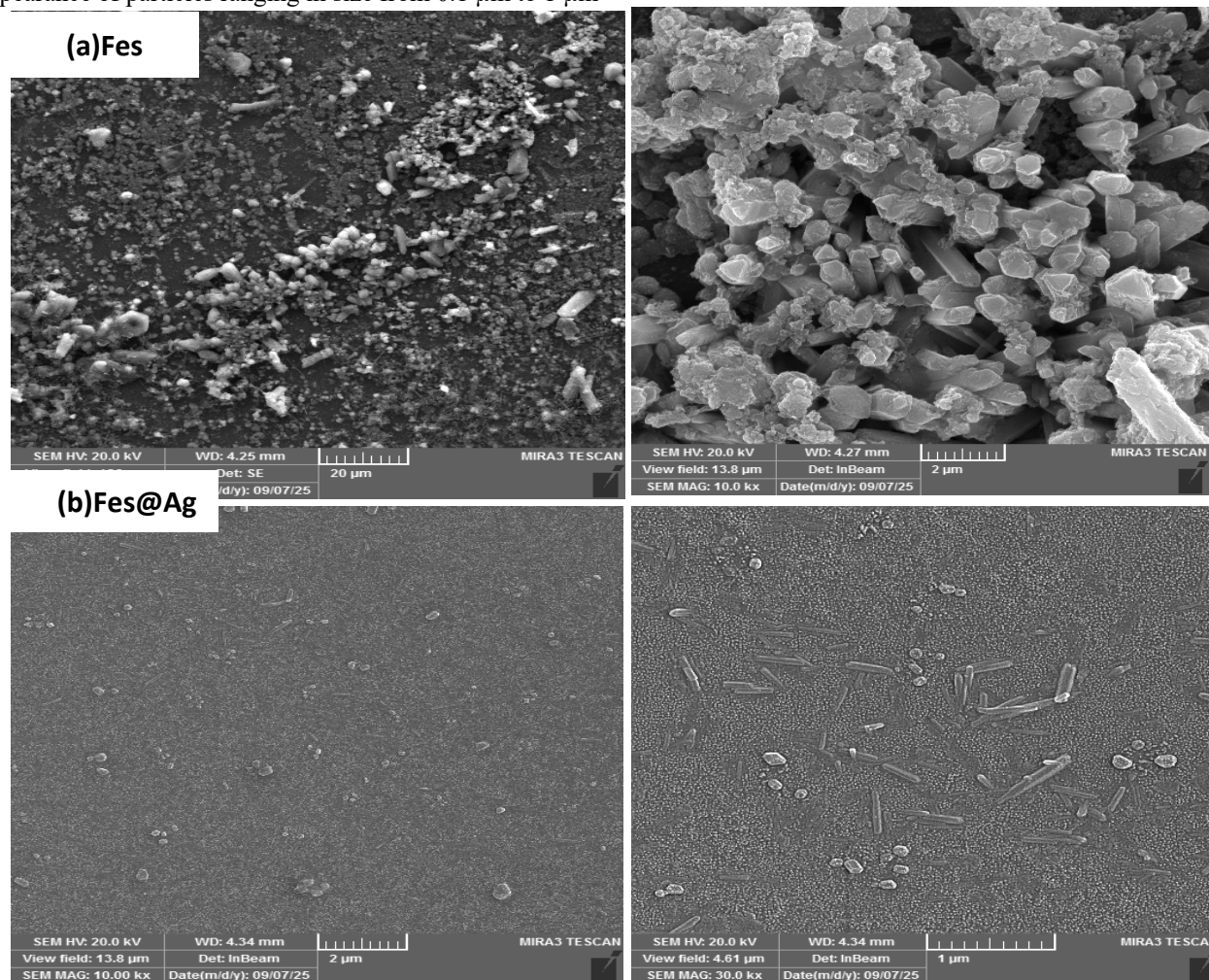


Fig. 3: scanning electrons microscopy images of (a)Fes (b)Fes@Ag

3.3 AFM Analysis

Figure (4)(a) shows the atomic force microscope (AFM) of a silver sulfide film, with the entire surface featuring white peaks of 40 nm and a roughness of 10-20 nm due to the prominent crystalline grains. Due to the use of the PLD method in this study and the effects of heat and pressure on this film with an average density, the film is not entirely homogeneous, as it contains both high- and low-nanograin areas. This indicates that the film was deposited in a way that clearly shows grain growth [28] [29] [30]. Although the

surface of the FeS@Ag film is depicted in Figure (4)(b), the deposition of silver particles on the FeS surface and their filling of the nano-gaps results in a noticeable drop in the RMS roughness value when compared to the pure FeS film, as reported in Table (3). While other researchers concluded that the prepared films were not homogeneous in this test because they used bases other than silicon, which may have been better bases, and because there was a difference in the concentrations of silver bombarded in the iron sulfide material, the presence of small particles distributed regularly

makes the surface more homogeneous and smooth [31].

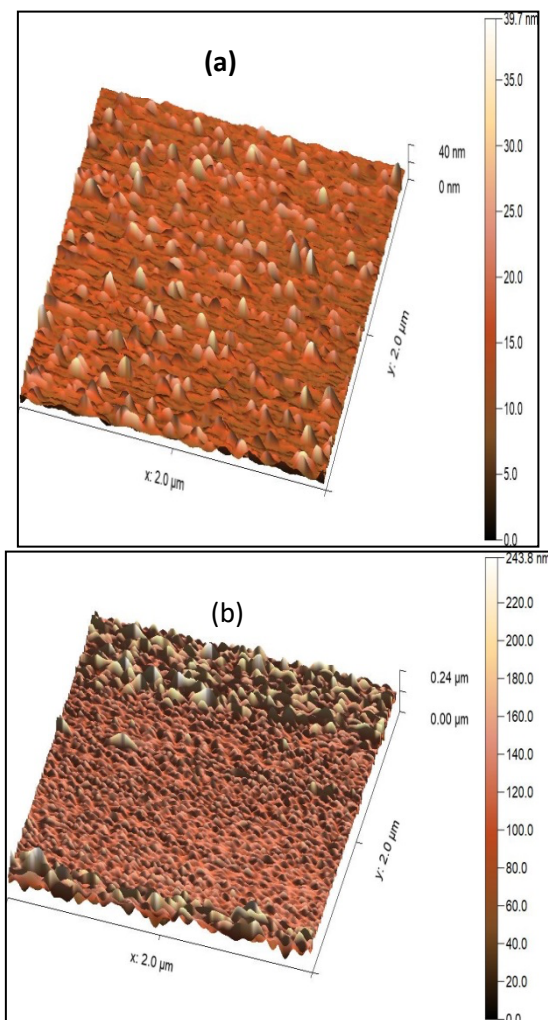


Fig. 4: Atomic force microscopy image of (a) FeS (b) FeS@Ag

Table 3: AFM Parameters

Samples	Average value (nm)	RMS roughness (Sq) (nm)	Mean roughness (Sa) (nm)
FeS	126.19	21.53	15.4
FeS@Ag	11.5	3.54	2.43

3.4 Optical properties

3.4.1 FTIR analysis

The bonds between the iron sulfide layer and the iron sulfide film under silver bombardment are depicted in Figure (5)(a). The poly saccharide C-O-C group and the stretching vibration of the (C=C) group cause two distinct peaks to emerge at wavelengths of 1120 and 1620 cm^{-1} , respectively. Because the film allowed these groups to absorb and show up in the FTIR, it was observed that the two peaks appeared significantly in the FeS film. The strength of these two peaks clearly decreased when the FeS@Ag film was used in figure (5)(b), demonstrating a number of possibilities, such as the

silver serving as a shield that lessens the exposed area carrying organic groups and other contaminants by preventing their adhesion to the exposed iron sulfide film. This suggests that the silver bombardment method was successful in this study, improving the surface film's purity and altering the chemical environment, which improved the film's performance [32]. This analysis concludes that the two films have stretching type single bonds, which results in low absorbance and relatively high transmittance. The UV-visible analysis supports this conclusion, as seen in figure (5). In contrast to this study, other researchers were able to perform a bond evaluation of the two materials developed in prior investigations, but no hydrocarbon linkages were discovered. This is explained by a number of factors, such as the employment of a pulsed laser without solvents or organic stabilizers and the dry preparation chamber with a high degree of vacuum, which prevents any carbon impurities that could alter the composition of the two materials [33].

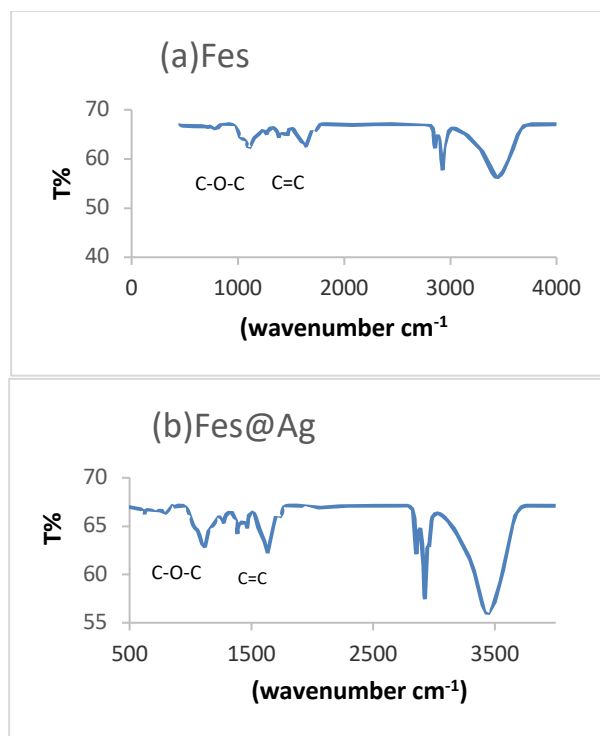


Fig. 5: FTIR image of (a) FeS (b) FeS@Ag

3.4.2 UV-Visible

The optical characteristics of the two films made using the pulsed laser approach, such as transmittance, absorbance, and energy gap, are examined in figure (5)(a)(b). It was discovered that the two films have comparatively high transmittance, which peaks at a wavelength of 900 nm. The FeS film has a relatively low absorbance and an energy gap of up to 2.5 eV, which is less than that of the FeS@Ag film. This suggests that the addition of silver particles to the FeS film raises the absorption at various visible wavelengths by generating new energy levels within the gap. However, because of the thin, semi-transparent structure, the

absorption is low and the transmittance is still very high. These films are therefore ideal for some medical applications, such as antibacterial medicines, because the absorbance does not reach particularly high values [34][35].

While the visual results of this investigation differed from those of other studies, as demonstrated in the study [36], this could be explained by different preparation techniques.

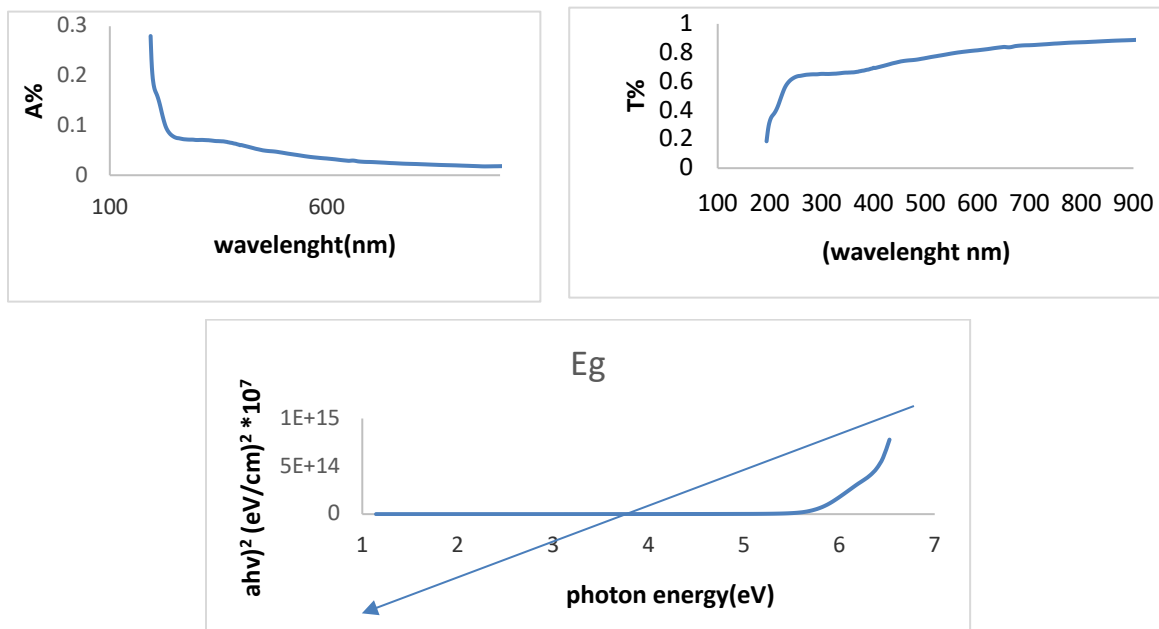


Fig. 6: (a) UV-Visible of Fes

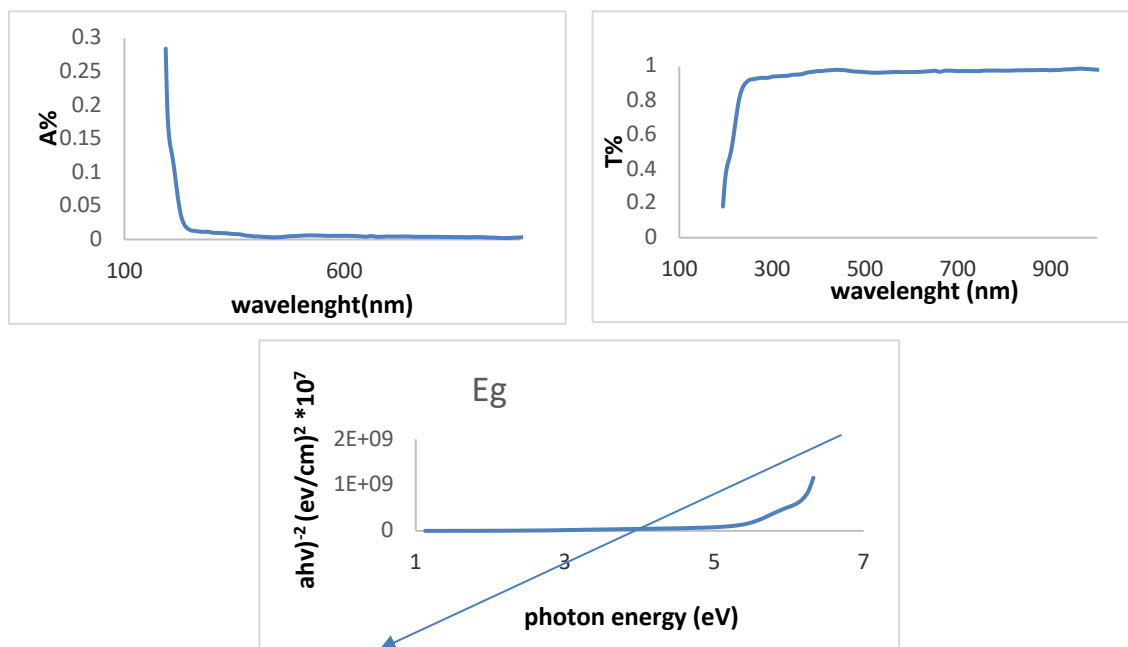


Fig. 6: (b) UV-Visible of Fes@Ag

The effect of Fes and Fes@Ag membranes on five different types of harmful bacteria is depicted in Figure 6. The (R1) Fes film had the greatest effect against *Staphylococcus aureus*, as indicated in Table 4, because it produced free radicals that damaged the cell wall and affected other harmful bacteria. Additionally, since silver has a highly toxic effect on bacteria and a strong ability to enter their cell walls, block their proteins, and prevent the formation of a biofilm

layer, it is evident that the (R2) Fes@Ag membrane enhances its efficiency against these hazardous bacteria. The two prepared membranes have very little effect on the bacteria of the types *E. coli* and *Candida albicans* because their walls are composed of two layers: a peptidoglycan layer with an outer membrane rich in lipopolysaccharides, which serves as a barrier to stop silver from entering the Fes@Ag film or the free radicals created by the Fes film and also has

antioxidant systems [37][38]. While other studies have demonstrated a significant impact of these two materials (FeS and FeS@Ag) on pathogenic bacteria of the E. Coli and Candida albicans types, the discrepancy in the findings

between this study and other studies may be caused by variations in the preparation techniques as well as the concentrations, composition, and particle size of the materials [39][40].

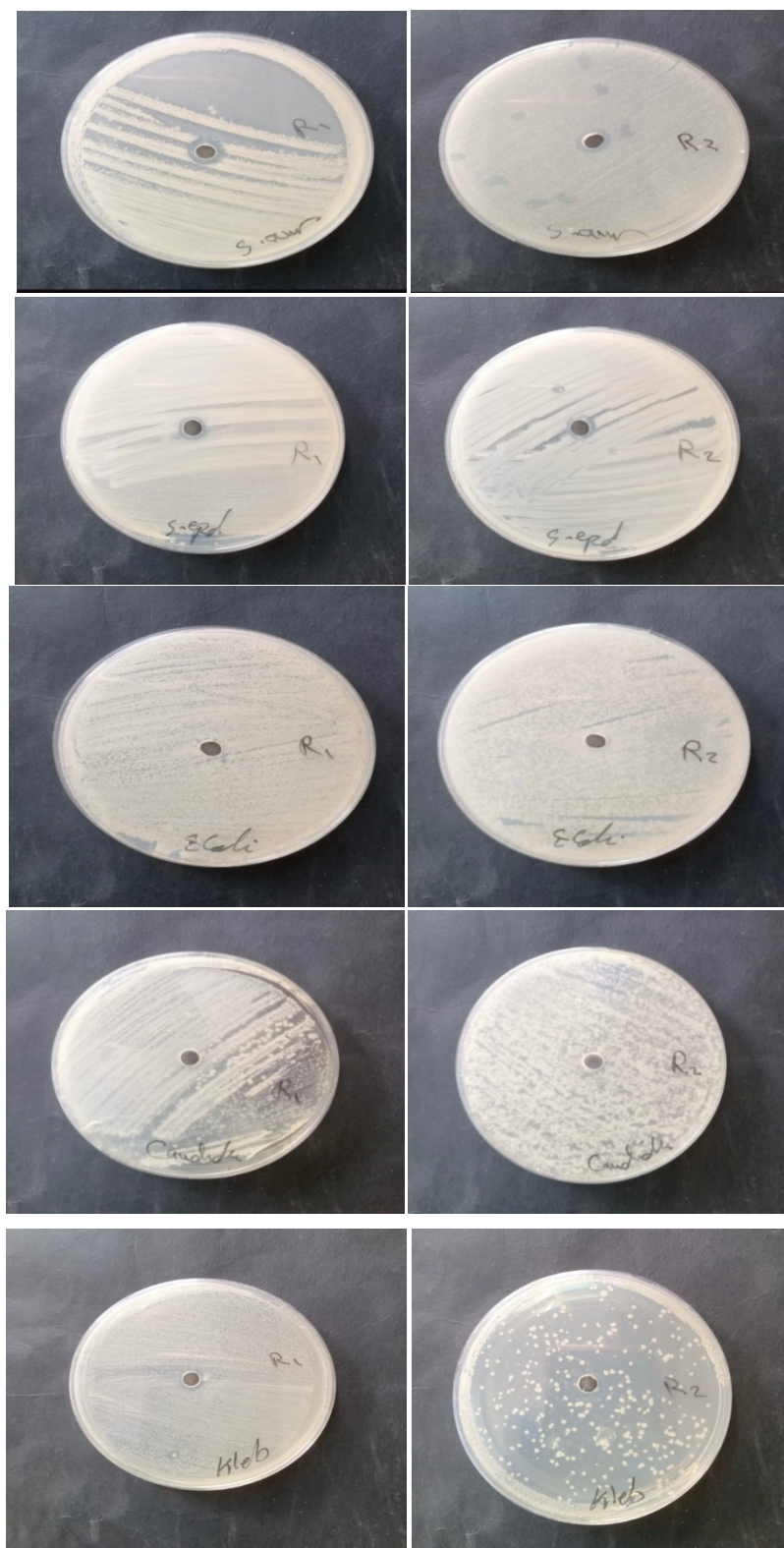


Fig. 7: biological activities of FeS and FeS@Ag against different type of bacterial

Table 4: the activity of antibacterial of Fes and Fes@Ag films

Bacteria's types	R1 Fes	R2 Fes@Ag
Staphylococcus aureus	13	10
Staphylococcus epidermis's	10	9
Escherichia coli	-----	-----
Klebsiella sp	10	12
Candida albicans	-----	-----

4 Conclusion

In this study, Fes film was created using the pulsed laser method, and another Fes@Ag film was created by bombarding it with silver. These advanced thin films are suitable for a variety of uses because the pulsed laser method is a contemporary, precise, and affordable technique. These two prepared membranes were selected to test their efficacy as antibacterial agents against five different types of bacteria that are dangerous to living things. Additionally, the membranes' capacity to prevent the growth of these dangerous bacteria makes them promising membranes as antidotes against dangerous types of bacteria.

Conflicts of Interest Statement

The authors certify that they have no conflicts of interest statement

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