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Investigation of the Effects of Different H_2SO_4, HCl, HNO_3 and $HClO_4$ Liquid Acid Media on the Synthesis of CdTe Semiconductor Thin Films for Solar Cells

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Abstract: The main target of the present paper is to investigate the effect of different acidic aqueous media(DAAM) on the synthesis of cadmium telluride thin films(CdTeTFm). The synthesis of CdTeTFm was carried out by the electrochemical deposition method(EDM) in DAAM. The chronoamperometry method of electrodeposition(ED) was used for the production of CdTeTFm. Furthermore, the electrochemical behaviors of the solutions were studied using cyclic voltammetry. The experiments were carried out with 3 electrodes (a working electrode (WE), a reference electrode(CE), and a counter electrode(RE)) using the electrochemical cell potentiostatic method. The experimental conditions of the acidic aqueous CdTe solution have been determined to be pH 3.56-3.57, the temperature of the solution is 85°C, the concentration of CdTe $2.45x10^{-1}$ M, and the reaction time is 25 minutes. The physical properties of CdTeTFm were determined by XRD, SEM/EDX, FT-IR, and UV-VIS analysis methods. According to the results of the analysis, it was observed that acidic aqueous media have an important role in the synthesis of CdTeTFm. The bandgap ranges and Cd/Te ratios of the synthesized thin films were obtained as 1.42, 1.48, 1.50, 1.58 eV, 0.65, 0.587, 0.79 and 0.738, respectively.

Keywords: CdTe; Solar cells; $HClO_4$; Electrochemical deposition method; Thin film.

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1. INTRODUCTION

The production of CdTe thin films has been very attractive because they can be useful in lasers, biosensors, photoelectric diodes, and protein markers, among other application fields (1,2) solar cells (SCs) (1-3). Moreover, CdTe cells have emerged as the most widely used commercialized thin film photovoltaic technology (4, 5). CdTe is now the only thin film technology in the top ten global producers. This is because CdTe is a very strong and extremely chemically stable material that can be deposited using a wide range of processes, making it perfect for large-scale production (5). Semiconductors (SmC) and nanocrystals (NCs) also have interesting electrochemical behavior. However, the applications in this area are currently limited by the low solubility of most of the NCs in watery media and the lack of a complete explanation of redox mechanisms (6-8). SmC devices based on thin films(such as CdTe) strongly depend on their structural and optical properties(OPs) (9,10). Moreover, the OPs of thin films depend on surface characteristics, shape, crystallite size, and other variables such as doping (11).

The highly efficient SC material "II-VI compound SmC," such as CdTeTFm is well known (12,13) and thought to be convenient due to its energy matching to the solar emitting spectrum and larger AC(absorption coefficient) natural structure (because of the direct transition energy band diagram and their values also are 1.45 eV and 1.5eV). It is almost optimum for photovoltaic SCs. Moreover, CdTe has a wide optical AC(> 105 cm^{-1}) compared to a-Si. So, something that has only a few microns of CdTe can absorb as high as 90% of photons (14).

Different growth methods, such as MBE(molecular beam epitaxy) (15) sputtering (16), ED (17-19), MOVPE (metal-organic chemical vapor deposition) (20) and chemical vapor deposition (21) have been improved. However, the ED of compound SmC thin films has some advantages. For example, (a) it is easy to check the electrical features of obtained films by managing deposition potential (DP) and adding impurities to the deposition films. (b) Because of the ease of device construction, it enables the production of wide-area films, and (c) it does not contain hazardous metal-organic reagents (for example, $Ga(CH_3)_3$; trimethylgallium). (d) It can be obtained at a low cost and with environmentally friendly energy. Furthermore, it was previously stated that 1.5 eV is the direct transition energy bandgap of CdTe (22). Moreover, that is the only material that can check the conduction sort and conductivity of II-VI materials by controlling impurity addition and exploiting ED.

Until now, the correlation between the electric features and the deposition conditions (which is the main carrier density (CD) and conduction sort) of CdTeTFm has been manufactured and explained openly by controlling the film composition and the DP. When the films are at a negative DP of more than -0.40 V versus Ag/AgCl on the positive part. In this case, CdTe is of the Te-rich p-type, while in the negative part, it is of the Cd-rich n-type (23). The CD of electro-deposited CdTeTFm is at most 10^{16} to 10^{17} cm^{-3} (24). On the other hand, it is too low to obtain high-efficiency SCs that require more than 10^{20} cm^{-3}. Thus, they researched the deposition method for CdTe thin films to control the transmission sort and CD, and stated the correlation between coating conditions and film features with Cu-doped CdTe (24, 25) where Cu works as a single acceptor. In the literature, it was observed that different methods carried out the synthesis of CdTeTFm. In this research, the synthesis of CdTeTFm was performed by EDM in different acids aqueous solutions, and with low pH values. Hence, a comparison of CdTeTFms obtained using four different acid solutions was made for the first time. Furthermore, one of the important points of this study, CdTeTFm, was obtained by the electrochemical deposition method with perchloric acid ($HClO_4$). $HClO_4$ is a chlorine oxoacid which is a water-soluble, colorless liquid.

In this work, CdTeTFm were obtained by using ED device of IVIUM VERTEX different acidic aqueous media. Furthermore, in the development of physical and chemical properties of synthesized CdTeTFm, mixing speed, duration, concentration, pH and temperature were investigated. Physical properties of obtained CdTeTFm were determined by XRD, SEM/EDX, FT-IR, and UV-VIS analysis methods.

Finally, according to the analysis (SEM, XRD, etc.) results, it was observed that synthesized CdTeTFm with $HClO_4$ acid is smoother than synthesized CdTeTFm with other acids (H_2SO_4 and HNO_3). Moreover, SEM images of CdTeTFm don't have pinholes on the surface.

2. EXPERIMENTAL

2.1. Synthesis of CdSe Thin Films

In this investigation, CdTeTFm was synthesized by using an ED device of IVIUM VERTEX in different acidic aqueous media. A 3-electrode ED cell was used for the deposition of $Cd(NO_3)_2$ containing ITO (indium-doped tin oxide as a WE), platinum wire (CE), and Ag/AgCl (RE). First, ITO-coated glass substrates (GSs) were washed with C_3H_6O and H_2O during a certain time to ensure that no pollution was left on the GSs. Second, Deposition baths consisted of 2.45×10^{-1} M $Cd(NO_3)_2$, Na_2TeO_3, 0.1 M KCl, and 3 drops of a solution of acid (HCl, H_2SO_4, $HClO_4$, and HCO_3). The cathodic potential was used to be -0.45eV. $Cd(NO_3)_2$ and Na_2TeO_3 were used as Cd and Te sources, respectively. H_2SO_4, HCl, HClO4, and HNO_3 acids were used to facilitate ion transfer in the solution medium. Synthesis conditions of CdTeTFm are shown in Table 1. CdTeTFm were called CdTe (HCl), CdTe (H_2SO_4), CdTe <($HClO_4$), and CdTe (HNO_3), respectively. When the bath temperature reached 85°C, the film was formed $Cd(NO_3)_2$ and Na_2TeO_3 covered on ITO-coated GSs during the depositions. Hence, analysis of current densities (CDs) time curves for different acidic aqueous media of samples was carried out using EDM for 25min. The experimental scheme is given in Figure 1.

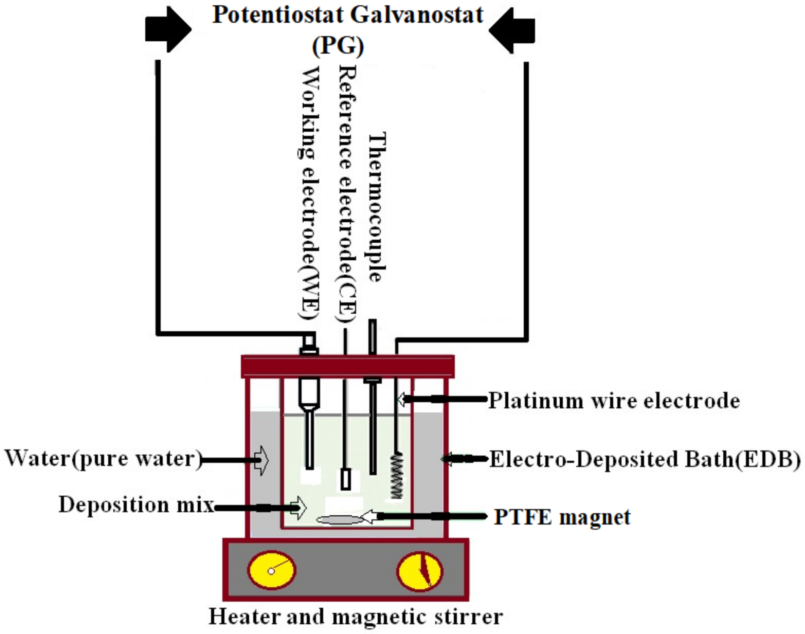

Figure 1: The experimental scheme of CdTeTFm.

Table 1: The synthesis parameters and chemicals of CdTe thin films.

Experiments	HCl	H_2SO_4	$HClO_4$	HNO_3
Concentration (M)	2.45x10^{-1} M	2.45x10^{-1} M	2.45x10^{-1} M	2.45x10^{-1} M
Cathodic potential (V)	-0.45	-0.45	-0.45	-0.45
Analysis time (min.)	25	25	25	25
pH	3.56	3.57	3.56	3.57
Acid type	HCl	H_2SO_4	$HClO_4$	HNO_3
Temperature (°C)	85	85	85	85
Source of Cd	$(CdNO_3)_2$	$(CdNO_3)_2$	$(CdNO_3)_2$	$(CdNO_3)_2$
Source of Te	Na_2TeO_3	Na_2TeO_3	Na_2TeO_3	Na_2TeO_3
Revolutions per minute (rpm)	800	800	800	800

2.2.Characterization studies

In this paper, FT-IR spectra of CdTeTFm were obtained in the range of 650-4000 cm^{-1}(the Perkin Elmer model IR). XRD analyses of CdTe also were performed using 0.066 step size, CuKα (λ=1.540 Å) radiation with 30V (tension), 40kV (current), and over the range 0°<2θ<70°(Panalytical Empryan HT-XRD). SEM and EDX mapping analyses were performed to obtain the surface morphology and the elemental composition of Cd and Te using the Zeiss SUPRA V 40. To determine the bandgap and adsorption wavelength features (OPs) of CdTeTFm was used, A JASCO V–530 double-beam UV–Vis spectrophotometer.

3. RESULTS AND DISCUSSION

Figure 1 illustrates UV-Vis analyses of the CdTeTFm. Hence, the OPs of CdTeTFm deposited versus acidic aqueous media were determined with A JASCO V–530 double-beam UV–Vis spectrophotometer. Band gaps of thin films have important effects on their performance such as solar cells. Therefore, the transmittance spectra of CdTeTFm are worth studying. It with different acidic aqueous media are given in Fig.2(b). It reports that CdTeTFm can absorb (Fig. 2(a)) almost all photons with a wavelength from 550 nm to 1000 nm (26).

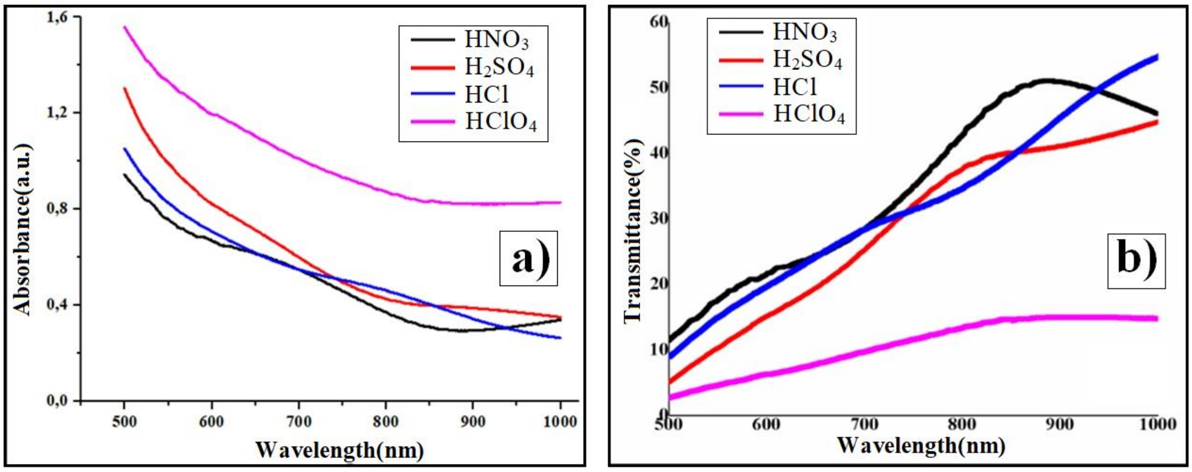

Figure 2: OPs of CdTe thin films deposited at versus acidic aqueous media a) Absorbance, b) Transmittance.

Figure 3 shows the CdTeTFm current densities. The current density depends on the thin film thickness and particle size (27).

At 85 °C, average CDs were obtained at 0.05 mA/cm^2. In addition, it was determined that the CDs increased proportionally by as much as 1.1 mA/cm^2 while the temperature value increased from 58 to 98°C. Therefore, it can be said that the reaction rate (RR) increases with temperature. It was observed that when the deposition temperature is increased, the molecular collision increases; thus, RR increases (28). Based on literature data, syntheses were performed at 85°C.

SEM images showed they agreed with the film particle sizes varying depending on the CD values (Fig. 3, 6 (a)). Besides the high CD values of HNO_3, the particles of CdTeTFm synthesized with HNO_3 were larger than the other thin films (Fig. 3, 6).

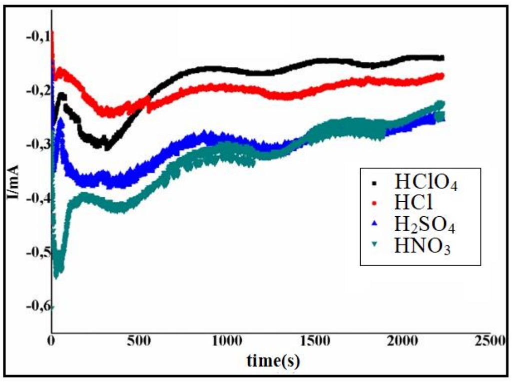

Figure 3: CD and time curves for different acidic aqueous media.

Figure 4 illustrates the XRD samples of synthesized CdTeTFm with diverse acidic aqueous media. The spherical-shaped grains structure of CdTeTFm was identified by the diffraction patterns between 2Θ = 20°−90° with main 5 peaks, which correspond to 2Θ=111,220, 311, 440, and 112 of ordered hexagonal structures. All CdTeTFm had a clear (d111, 220 and 311) CdTe main Bragg peaks occurring at 2θ=23.72°, 30.51°, 39.22°, 45.20°, 55.52° and 59.89°(29). The crystallite sizes corresponding to different aqueous media were calculated using the Scherrer equation (28, 30).

$$\boldsymbol{D} = \frac{k\lambda}{B(radian)cos(\theta_B)} \quad (1)$$

In the above expression of the Scherrer equation, "D" (the average size of the crystallites), particle shape factor "k" was taken to be 0.94, the wavelength of the X-ray is "λ" (λ= 0.1542 nm), "B" is the FWHM intensity, and ΘB is the Bragg angle. To determine the average nanoparticle crystalline size of CdTeTFm, the values associated with the 2Θ=$d_{(111)}$ plane in the Scherrer equation were replaced.

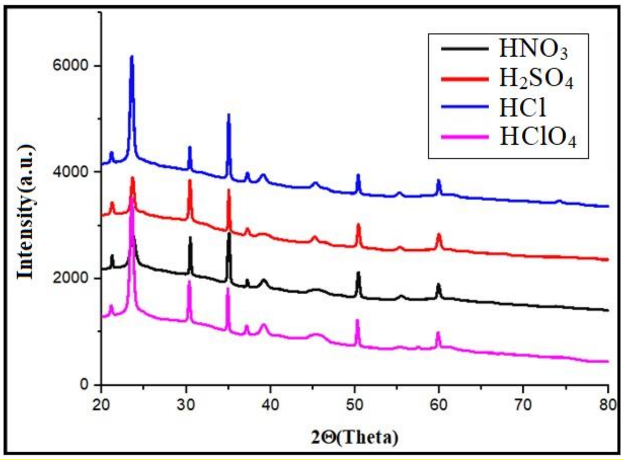

Figure 4: The XRD spectra of CdTeTFm deposited under different acid solutions HNO_3, H_2SO_4, HCl and $HclO_4$.

Despite very few shifts in the XRD d(111) main peaks, the crystallite sizes and FWHM(°) values of all thin films were 35.66 and 0.2378, respectively (Table 2 and Fig. 4). However, film thickness affects the intensity of XRD peaks. Furthermore, XRD peaks of thick films have a low intensity (Fig. 4, d(111)). However, if thin films have high sensitivity, these films are said to crystallize very well. When well-crystallized films are used as a permeable layer in solar cells, light can pass easily. This facilitates the passage of the photons between "n" and "p" regions. It allows the formation of electron-hole pairs. Moreover, it is preferred for solar cell applications.

Optical absorption measurements of thin films coated on glass were made. The photon energy, depending on the absorption coefficient, was calculated with the Tauc formula and can be expressed as follows:

$$\alpha hv = A(hv - g)n \qquad (2)$$

The graphs of the thin films were drawn (Figure 4), and the band gaps were determined (Table 2). Here, the photon energy is hv, the absorption coefficient is α, A constant, and Eg band gap, n=1/2 for the direct band gap. The "n" of the indirect band gap, on the other hand, is assumed to be 2 (n = 2).For thin films, it also depends on the thickness of the film(31,32). The direct transitions CdTeTFm are used(29).

The calculated band gaps of CdTe ($HclO_4$, HNO_3, H_2SO_4 and HCl) thin films were obtained as 1.42, 1.48, 1.58, and 1.50 eV, respectively (Figure 3 and Table 2). As expected, CdTe($HclO_4$) thin films with small particle sizes have been determined to have lower forbidden energy band gap values. This situation can be explained by the relationship between matter and the light used.

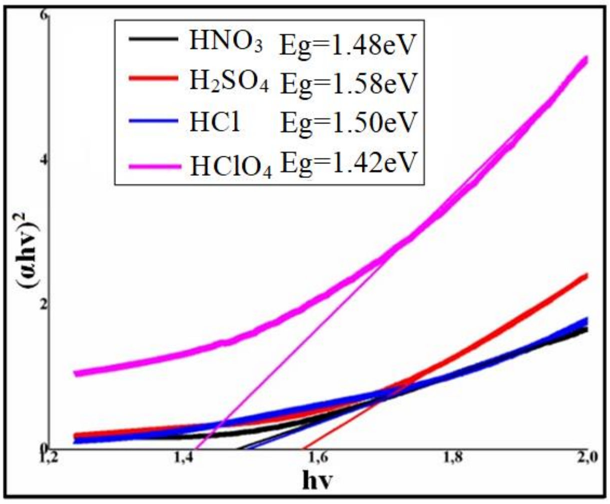

Figure 5: Tauc's plot curves of CdTeTFm.

Table 2: Physical properties of CdTeTFm.

Experiments	HNO_3	H_2SO_4	HCl	$HClO_4$
Crystallite Size(nm)	11	20	20	17
FWHMβ(°)	0.2378	0.2378	0.2378	0.2378
Band gap(eV),(Tauc's plot)	1.48	1.58	1.50	1.42

FWHMβ(°) (full width at half-maximum).

Figure 6 shows the SEM images of the CdTeTFm (30kx magnifications). The surface morphology results of CdTeTFm showed different surface morphologies in different acidic aqueous media. Nitric acid-synthesized CdTe thin films had more clear spherical shaped grains (25) and were larger than others (Figures 6 and 7(a and c)). Furthermore, as the acidity of the solution decreased, the surfaces of the synthesized thin films became smooth (Fig. 7 (a, c)). According to EDX analysis results, Cd/Te ratios of CdTe thin films synthesized in different aqueous media were obtained as 0.65, 0.587, 0.79, and 0.738, respectively (Fig. 6 and 7(b and d)).

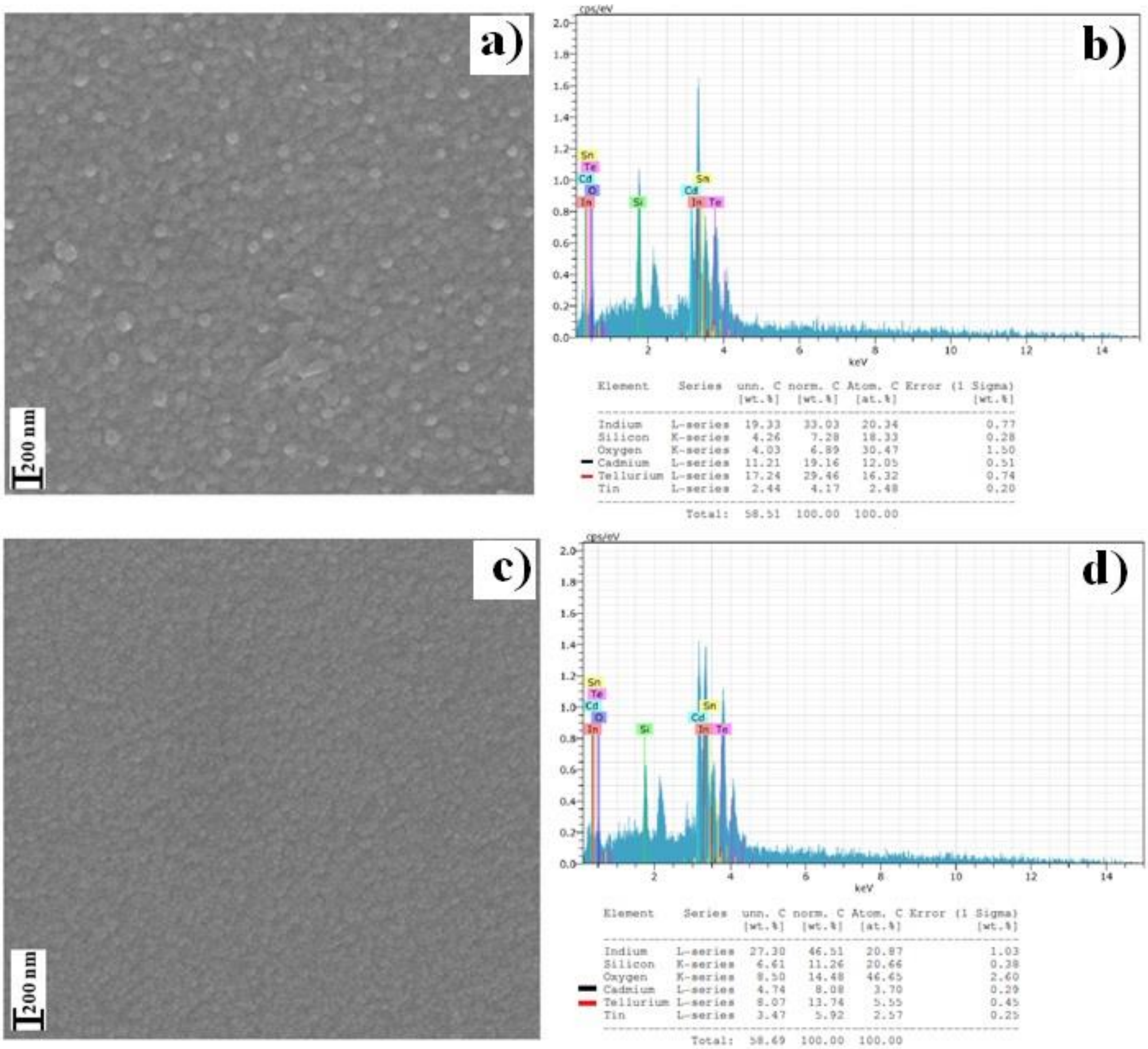

Figure 6: SEM images and EDX results of CdTeTFm deposited under different acid solutions (a, b) HNO_3, (c,d) H_2SO_4.

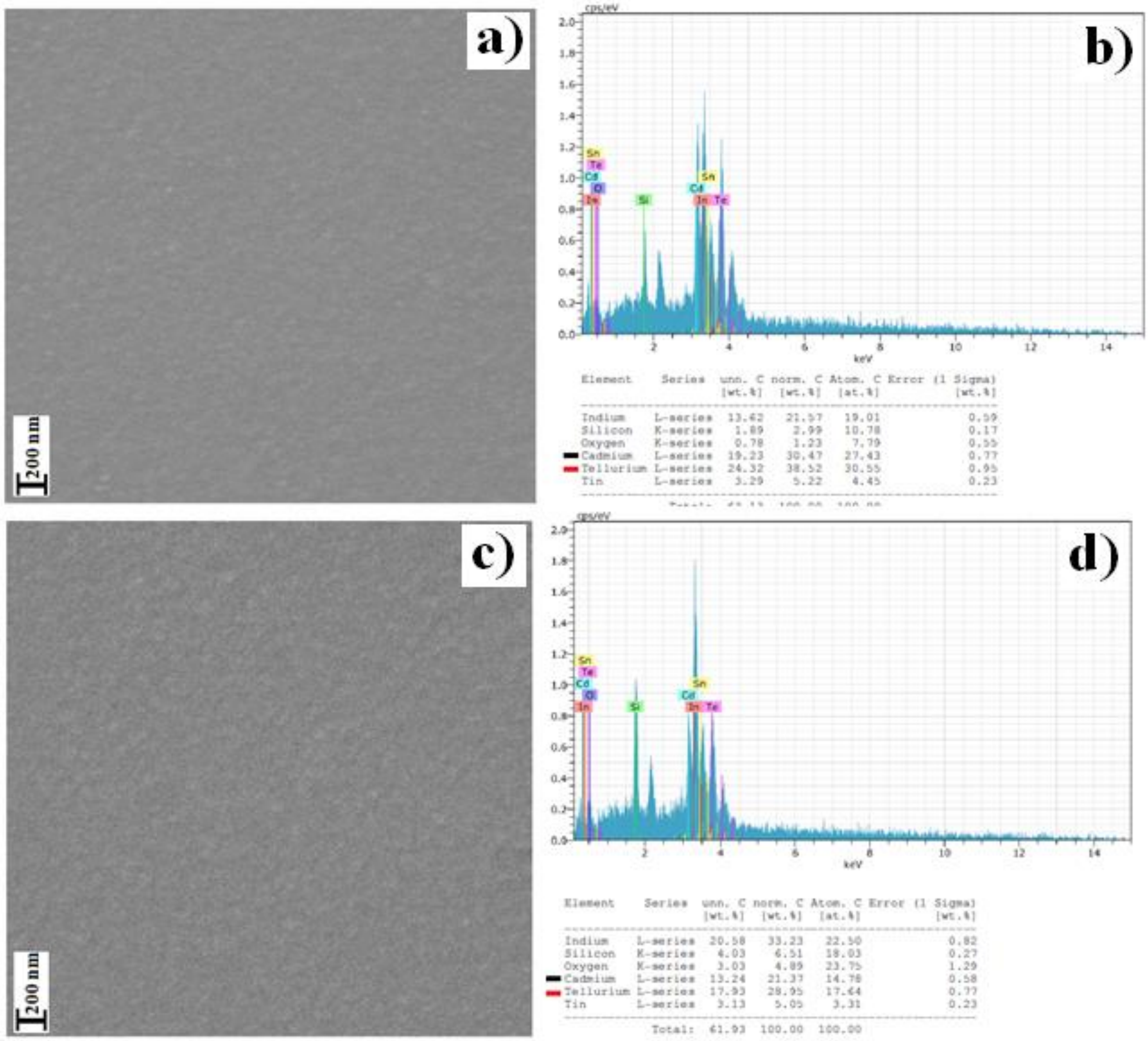

Figure 7: SEM images and EDX results of CdTeTFm deposited under different acid solutions (a, b) HCl, (c, d) $HClO_4$.

The thick films are not desirable in solar cells. However, the high absorbance of the films is desirable if it can be applied to gas sensors. The flatness of the obtained thin films does not scatter the photons. That is, this property increases the permeability of the films. If the surfaces of the synthesized films are rough, they cause the scattering of photons, reduce permeability, and increase absorption. Moreover, thin films to be used in solar panels should have an optimum 1.5eV and high permeability and absorption values. These properties can be obtained as a result of easier penetration of rays into the P semiconductor region.

In addition, surface characterizations of the obtained thin films were determined with the ImageJ(Fiji-win64-Fiji.app) program. The surface plot, 3D surface plot, and SurfChar1 q programs were used for the surface morphologies of the thin films. The results obtained are given in Figure 8. According to SurfCar1 q analysis results, roughness values were obtained as 11.9880, 10.6749, 8.6264, and 6.9208nm, respectively. It has been observed that these results are compatible with surface plots and 3D surface plot images. It was determined that the thin film synthesized with HCl was less rough and the grain sizes were more homogeneous (Figure 9).

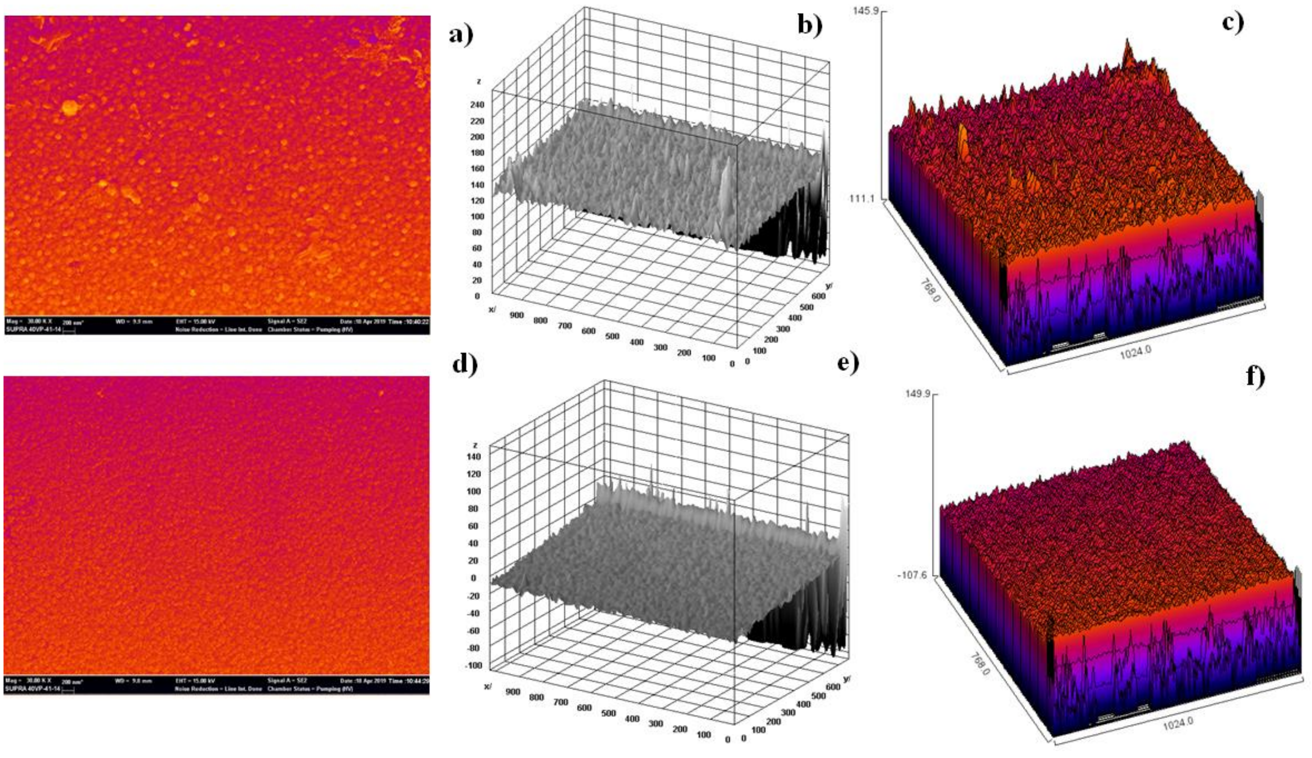

Figure 8: ImageJ analyses of CdTe (a, b, c are HNO_3 and d, e, f are H_2SO_4).

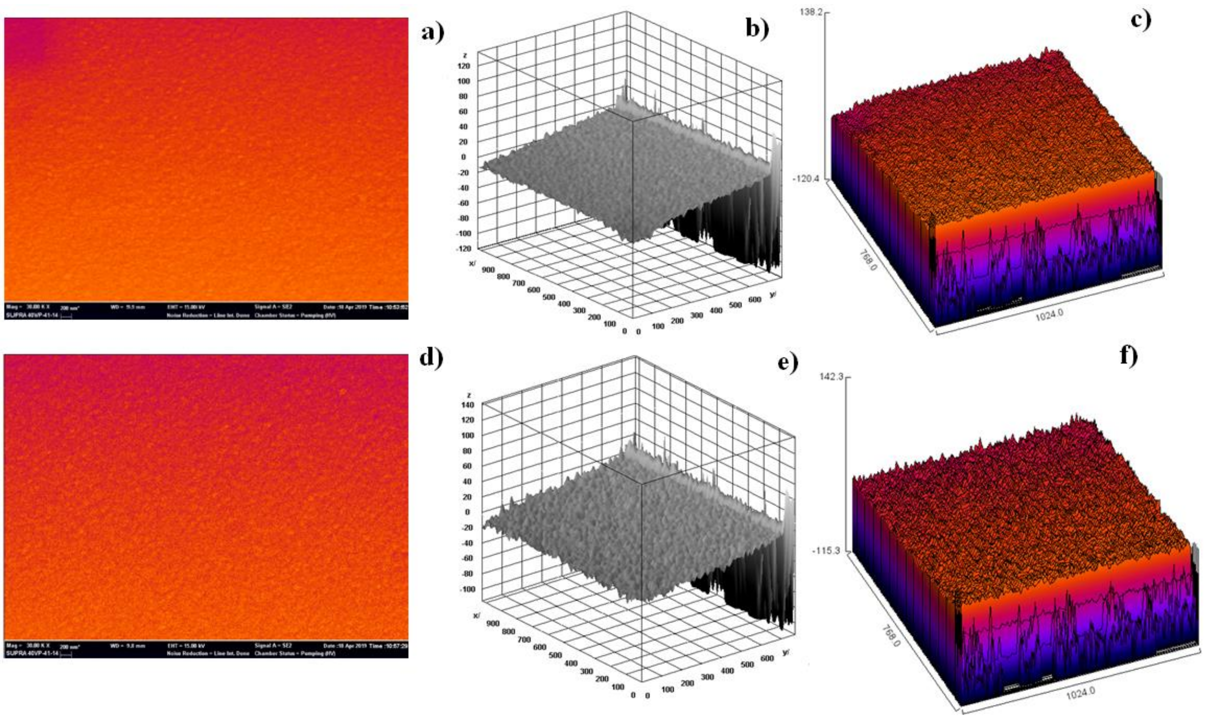

Figure 9: ImageJ analyses of CdTe (a, b, c are HCl and d, e, f are $HClO_4$).

4. CONCLUSION

CdTe thin films were obtained by the electrochemical deposition method. The effects of DAAM(such as HCl, H_2SO_4, $HClO_4$, and HNO_3) on the film surface, the structural morphology, and the OPs of the thin films obtained were investigated by XRD, SEM/EDX, FT-IR, and UV analysis methods. Moreover, a novel investigation of synthesized CdTeTFm with $HClO_4$ acidic aqueous media has been reported in the literature. By investigating the OPs of semiconductors, information regarding the behavior of electrons and holes in the material, as well as band structures, can be gleaned. Each material emits varied and distinct wavelengths. Similarly, the wavelengths absorbed by each substance will differ from those of other materials. The amount of light absorbed can have positive effects on optical properties. Therefore, the surfaces, shapes, and crystal sizes of the synthesized thin films are important. It was observed that SEM images of CdTeTFm obtained with nitric acid were rough compared to other films. However, no pinholes were observed on the film surfaces. The permeability of the beams, which is thought to be due to the film thickness, was low. Despite the low transmittance of $HClO_4$, the current density and absorbance value were higher than other thin films. It was observed that the surface of the CdTe thin film synthesized with $HClO_4$ was smoother than the films obtained with HCl and H_2SO_4, and also that the Cd/Te ratio was higher. This result is predicted to be due to the grain size thickness of CdTeTFm synthesized with $HClO_4$. We think that since $HClO_4$ has a higher acidity than other solvents, it causes thinner smooth thin films to be obtained. The grain size and film thickness of $HClO_4$ were calculated as 105 and 344 nm, respectively. Moreover, it has the lowest forbidden band gap. Solar cells can store energy more efficiently with thin films of low transmittance and high absorbance. As a result, it has been seen that CdTeTFm prepared with $HClO_4$ is a good candidate for solar panels with the lowest transmittance and highest absorbance value.

5. CONFLICT OF INTEREST

The authors confirm that this article's content has no conflict of interest.

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