Süleyman Demirel Üniversitesi
Fen Bilimleri Enstitüsü Dergisi
Cilt 22, Sayı 1, 134-140, 2018

Süleyman Demirel University
Journal of Natural and Applied Sciences
Volume 22, Issue 1, 134-140, 2018

DOI: 10.19113/sdufbed.30879

Modification of Morphological, Structural and Optical Properties of CBD-Based Growth of PbS Films on Glass Substrates by Addition of Saccharin

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(Alınış / Received: 13.10.2017, Kabul / Accepted: 29.01.2018, Online Yayınlanma / Published Online: 17.03.2018)

Keywords
Surface roughness,
Lead sulphide,
Saccharin,
Chemical bath deposition

Abstract: Saccharin which so-called as benzoic sulfimide is an artificial sweetener. In this paper, nanostructured lead sulfide (PbS) thin films were produced in the presence of additive known as saccharin via chemical bath deposition (CBD). Effect of the saccharin content on the morphological, optical and structural properties of the films has been investigated. Lead sulfide (PbS) films that included additive were prepared at five different concentrations of saccharin (from 0 to 5%). The fabricated samples were characterized by surface roughness, scanning electron microscopy (SEM), X-Ray diffraction and UV-vis spectroscopy. It was found that the presence of saccharin highly affected the optical and structural properties of PbS thin films. According to the X-Ray diffraction results, crystallite size values of the films decreased from 12.88 to 5.25 nm with increasing of saccharin content. UV-vis measurements showed that both the band gap and transmission properties of the films increased as a result of increasing saccharin concentration. Further, Surface roughness measurements exhibited that average surface roughness was diminished with increasing saccharin content. As a result, it has been found that the presence of saccharin in growth solution played a significant mission in adjusting the main physical properties of the samples.

Sakarin İlavesiyle Cam Altlıklar Üzerine CBD ile Büyütülen PbS Filmlerin Morfolojik, Yapısal ve Optik Özelliklerinin Modifikasyonu

Anahtar Kelimeler
Yüzey pürüzlülüğü,
Kurşun sülfür,
Sakarin,
Kimyasal banyo depolama

Özet: Benzoik sülfimid olarak da bilinen sakarin yapay bir tatlandırıcıdır. Bu makalede, nanoboyutlu kurşun sülfür (PbS) ince filmler sakarin olarak bilinen katkı maddesi varlığında kimyasal banyo depolama (CBD) ile üretilmiştir. Sakkarin miktarının filmlerin morfolojik, optik ve yapısal özelliklerine etkisi araştırılmıştır. Katkı maddesi içeren kurşun sülfür (PbS) filmler sakarinin beş farklı konsantrasyonunda (%0 dan %5'e) hazırlanmıştır. Üretilen numuneler yüzey pürüzlülüğü metodu, taramalı elektron mikroskopisi (SEM), X-Işını kırınımı ve UV-vis spektroskopisi ile karakterize edilmiştir. Sakarin varlığının PbS ince filmlerin optik ve yapısal özelliklerini oldukça etkilediği bulunmuştur. X-Işını kırınımı sonuçlarına göre, filmlerin kristal boyutu değerleri sakarin içeriğinin artmasıyla 12.88 nm'den 5.25 nm'ye düşmüştür. UV-vis ölçümleri artan sakarin konsantrasyonunun bir sonucu olarak hem filmlerin bant aralığının hem de geçirgenlik özelliklerinin arttığını göstermiştir. Ayrıca, yüzey pürüzlülüğü ölçümleri sakarin miktarının artmasıyla ortalama yüzey pürüzlülüğünün azaldığını göstermiştir. Sonuç olarak, büyütme çözeltisinde sakarin varlığının numunelerin temel fiziksel özelliklerini ayarlamada önemli bir görev üstlendiği tespit edilmiştir.

1. Introduction

Metal chalcogenide films have received great interest in recent years because of their use as semiconducting materials for solar cell applications [1]. Preparation of polycrystalline metal chalcogenide films can be made inexpensively and simply [2]. Among the various semiconductor nanostructures, lead sulfide which is an important member of the chalcogenides has high absorption coefficient, high photosensitivity and high carrier mobility [3]. Also lead sulphide desirable and attractive for various

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applications such as infrared detection [4] photovoltaic applications [5] and biosensors [6] because of its high chemical stability, excellent photosensitivity, a large exciton Bohr radius (18 nm), wide range band gap variation and abundance in nature. Also, lead sulfide (PbS) which is a p-type semiconductor material can use as a buffer layer [7].

Various methods were used to deposit PbS chalcogenide films including CBD [2], sol-gel [8], electrodeposition and SILAR etc. [9]. Among techniques used to fabricate PbS thin films, CBD offers many advantages such as easy processing, quick, low temperature and little cost. Crystal structure, surface morphology and particle size which are all influenced by processing conditions and bath composition, have a significant effect on the photovoltaic behaviour of the nanostructures. It is mentioned in the literature that during the growth process, it is possible to modify and control the surface morphology and the crystalline size with the use of additives [10, 11].

Generally, it is known that additive agents are added to the reaction medium to enhance the general quality or to counteract undesirable properties. Saccharin which is an organic chemical compound is one of the additives mentioned in the literature. It is known that saccharin which so-called as benzoic sulfimide is an artificial sweetener. Saccharin has been used as an additive agent in the reaction medium in order to improve the characteristics of nanoparticles [10-12]. We aimed in this submission to show the influence of saccharin level on the crystal structure, particle size, optical properties and surface roughness of the PbS thin films. Thus, we have decided to use the saccharin as an additive material in the growth process due to the potential to enhance the physical properties of PbS nanostructures of the saccharin. According to our knowledge, there is no report so far about the effects of saccharin on the surface roughness, morphological, structural and optical feature of lead sulfide (PbS) films. In order to understand characteristic properties of PbS films produced by saccharin addition, we investigated the surface roughness, optical, structural properties and surface morphology of the PbS coatings as a function of saccharin level in this novel research.

2. Material and Method

2.1. Substrate cleaning

In the study, PbS films produced by saccharin addition and pure PbS films were coated on commercial quality glass substrates (75 mm x 25 mm x 1 mm) by CBD method. Prior to the deposition process, the substrates were cleaned with distilled water, acetone and dilute HNO_3 solution (20% v/v) for 10 min respectively. Then, the glass substrates were washed again with deionized water and dried with a hair drier prior to a deposition process.

2.2. Sample preparation

All chemicals used in the experiment were analytical grade and purchased from Merck. Deposition of the samples (pure or containing saccharin) was done in a beaker which is included the reactive solutions. The reactive bath containing 0.5 M Lead (II) acetate trihydrate ($Pb(CH_3COO)_2.3H_2O$), 1M thiourea (NH_2CSNH_2), 1M triethanolamine ($C_6H_{15}NO_3$) and 1M tri-sodium citrate ($C_6H_5Na_3O_7$) was used for the deposition of PbS films. The pH of the reactive solution was fixed at 12.5 using NaOH solution. In order to obtain the saccharin containing baths, the main solution was poured into six different beakers and as the required amount of additive reagent (0, 1, 2, 3, 4 and 5 at. % of saccharin) was added to the starting solutions. Soda lime glass slides were immersed as vertical in the chemical solution at 303K and the substrates were removed from the solution after the deposition time of 18 h as reported elsewhere [2]. After the deposition process, the colour of the chalcogenide films was dark gray and their appearance was like mirror. The reaction mechanism for the fabrication of PbS films is as follows [5]

$$Pb(AC)_2.3H_2O + TEA \rightleftarrows Pb(TEA)^{2+} + 2(AC^-) \quad (1)$$

$$Pb(TEA)^{2+} \rightleftarrows Pb^{2+} + TEA \quad (2)$$

$$[(H_2N)_2CS] + OH^- \rightarrow CH_2N_2 + H_2O + HS^- \quad (3)$$

$$HS^- + OH^- \rightleftarrows H_2O + S^{2-} \quad (4)$$

$$Pb^{2+} + S^{2-} \rightarrow PbS \quad (5)$$

2.3. Sample characterization

Rigaku Smart Lab model X-ray diffractometer was used to determine the crystal structure of the films. Surface morphology of pure and saccharin-added samples was studied using EVO40-LEO model SEM. Measurements of surface roughness (Ra value) of the fabricated films was made by using a Surface roughness tester (Taylor Hobson Surtronic 25). Optical properties such as band gap and transmittance were measured using Thermo Scientific Evolution 160 UV-visible spectrophotometer.

3. Results

3.1. Structural properties

XRD patterns of lead sulfide (PbS) nanostructures grown at different molar concentrations of saccharin are shown Figure 1. The pattern of pure PbS thin film (without saccharin) was also illustrated in the figure.

For PbS chalcogenide films, there were the peaks at 2θ≈25.90°, 30.00°, 42.95°, 50.90°, 53.33°, 62.43°, 68.85°, 70.83°, 78.78° and 84.69° corresponding to the reflected planes of (111), (200), (220), (311), (222), (400), (331), (420), (422) and (511) of lead sulphide. XRD patterns of the nanostructures indicated that the samples are polycrystalline with cubic structure (JCPDS: 05-0592). The main peaks at 25.90° and 30.00° corresponding to (111) and (200) planes proves the preferred crystallization. Other observed peaks are less intense as compared to (111) and (200). Also, the XRD patterns do not show any other peaks related to impurity. As can be seen from XRD results that the intensity of the peaks corresponding to (111) and (200) planes reduces with increasing the concentration of saccharin in the growth solution.

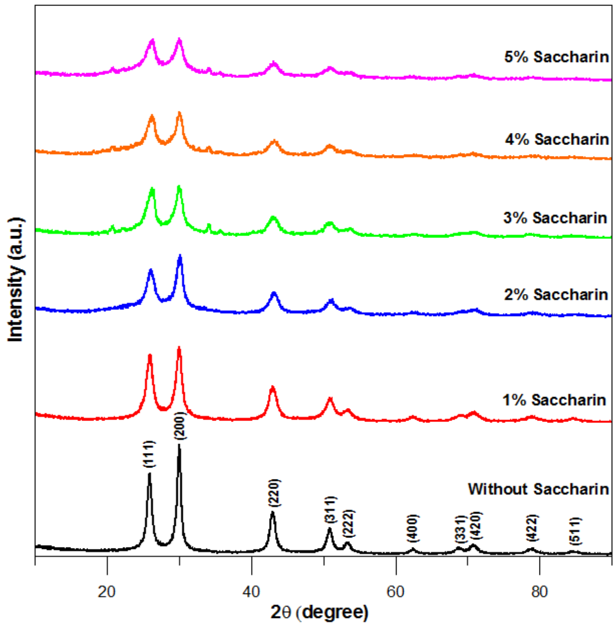

Figure 1. X-ray diffraction patterns of the PbS films as a function of Saccharin amounts in the growth baths.

This result can be attributed to additive induced some structural disorder in the films. Moreover, the broadening of the peaks was observed with increasing saccharin level in the growth bath. The reason for the broadening may be associated with the reduce in the crystalline size. The decrease of crystalline size may result in an increase in the grain boundaries and so increase the amount of defects in the structure [2, 13]. The recorded (200) and (111) peak intensities of the coatings are shown in Table 1.

Texture can be explained as the distribution of crystallographic orientations of a polycrystalline sample. The values of the texture coefficient $T_{C(hkl)}$ for four different planes are shown in Table 1. The addition of additive agent in the bath may induce the change of diffusion rate of cationic and anionic precursors at the surface during coating operation and this behaviour leads to a change of preferred orientation. In order to investigate a probability of the preferred orientation, texture coefficients corresponding to (111), (200), (220), and (311) planes have been calculated in all the films from the XRD spectrum according to the following formula [14].

$$TC_{(hkl)} = \frac{I_{(hkl)}/I_{0(hkl)}}{N^{-1}\sum_N I_{(hkl)}/I_{0(hkl)}} \qquad (6)$$

where $I_{(hkl)}$ is the measured intensity of *hkl* plane, $I_{0(hkl)}$ is the intensity of *hkl* plane on a completely random sample taken from a powder diffraction file (PDF) card, and N is the number of diffraction peaks considered in the analysis. The (200) plane has the highest value (1.65) of texture coefficient $TC_{(hkl)}$ for the whole films. This result indicates that majority of the grains are crystallized in (200) plane. It is known that if texture coefficient is equal to 1 or less than 1 the crystallites are considered to be randomly oriented [14]. From the Table 1, it can be seen that for all the films texture coefficients along (200) and (111) planes are maximum and more than 1, whereas for other planes it is either less than 1. We can say that (200) plane is the preferred orientation of all the samples.

Table 1. Surface roughness parameters (Ra), recorded peak intensity and $T_{C(hkl)}$ values of the PbS coatings as a function of Saccharin content.

Saccharin concentration in the growth solution %	Ra^a (μm)	RSD (%) $n = 3$	Recorded Peak Intensity		$T_{C(hkl)}$			
			(200)	(111)	(200)	(111)	(220)	(311)
0	0.56±0.01	1.79	2151	1605	1.65	1.23	0.68	0.43
1	0.48±0.02	4.17	1497	1357	1.45	1.31	0.73	0.51
2	0.40±0.02	5.25	1250	972	1.58	1.23	0.68	0.52
3	0.36±0.03	7.06	1058	1033	1.44	1.41	0.65	0.50
4	0.30±0.02	6.67	969	924	1.44	1.37	0.66	0.53
5	0.28±0.02	5.52	882	854	1.41	1.36	0.70	0.52

[a] Average of three values ± Standard deviation , RSD: relative standard deviation

The crystallite size of the coated samples is calculated via Scherrer equation [13].

$$D = 0.94\,\lambda/\beta cos\theta \qquad (7)$$

where λ is the wavelength of X-ray radiation, θ is the Bragg angle of the peak and β is the full width and half maximum (FWHM) of the diffraction peak in radians. FWHM, crystallite size, dislocation density, band gaps and microstrain value of the samples as a function of saccharin level are listed in Table 2. From the Table, we can observe that crystallite sizes of the coatings decreased from 12.88 nm to 5.25 nm with increasing of additive level from 0% to 5%. The existence of saccharin concentration in the growth bath may affect nucleation process and consequently the number of nucleation sites leading to the formation of smaller crystals [15, 16].

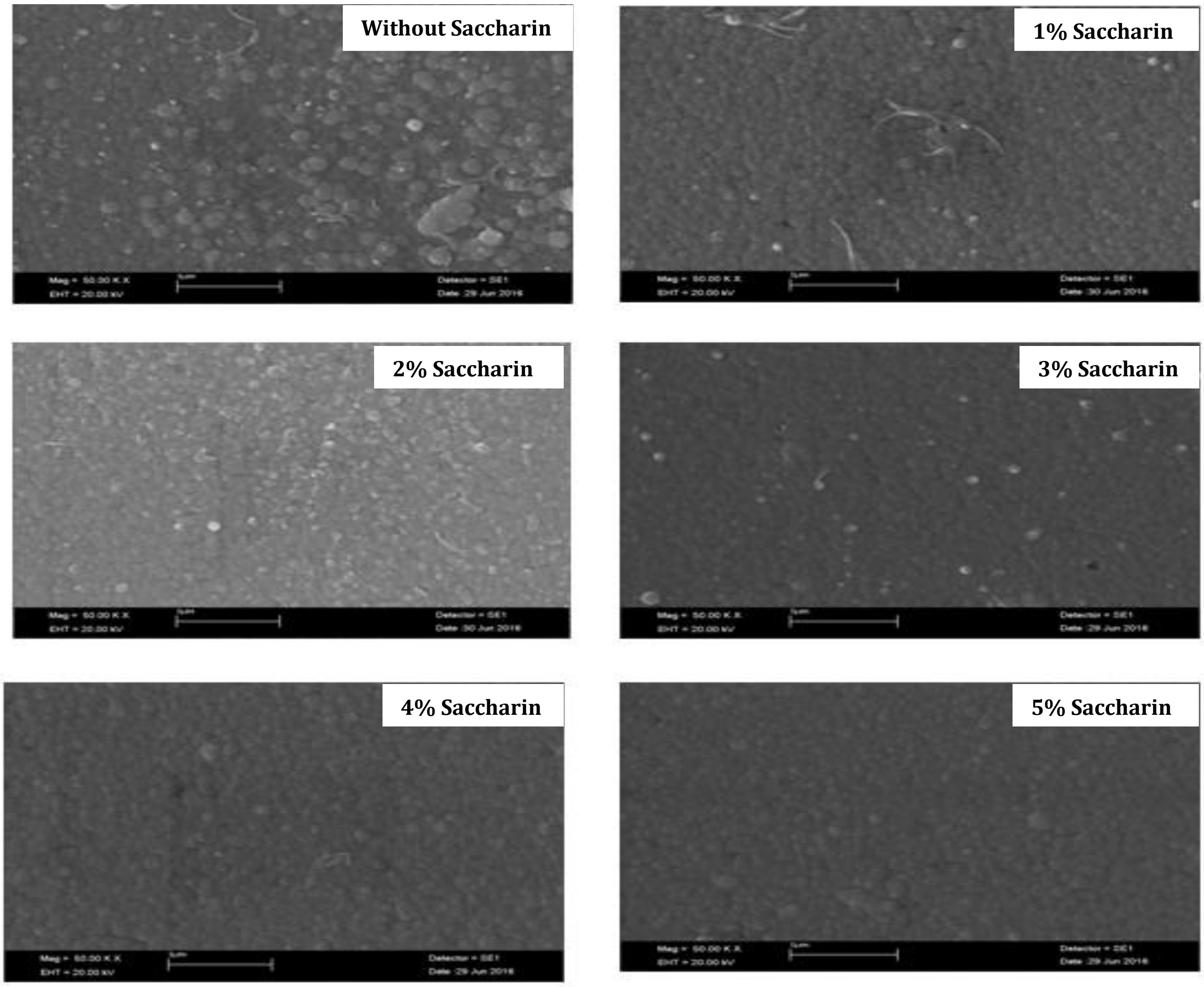

Figure 2. SEM micrographs of the PbS samples as a function of Saccharin levels in the baths.

Microstrain and dislocation density (ρ) values of the samples are determined using the formulas given below [13];

$$\varepsilon = \beta cos\theta/4 \tag{8}$$

and

$$\rho = 15\varepsilon/aD \tag{9}$$

where D is average crystallite size and a is the lattice parameter.

According to Table 2, microstrain values of the samples increased when saccharin amount was increased from 0 to 5%. A Similar trend was also observed for the dislocation density of the PbS thin films. Both behaviour of microstrain and dislocation density values of the PbS has reverse dependence with the crystallite size values as expected. It is known that the presence of any defect in a structure such as micro strain, dislocations, crystallite size, vacancies, vacancy clusters and residual internal stress lead to the excess free volume at the grain boundaries. The decrease of crystalline size may result in an increase in the grain boundaries and so increase the amount of defects in the structure [17].

Table 2. FWHM, crystallite size, thickness, microstrain, dislocation density and band gaps of the PbS coatings as a function of Saccharin level.

Saccharin concentration in the growth solution %	FWHM (radian)	Crystallite size (*D*) (nm)	Thickness (nm)	Microstrain (Ɛ) x 10^{-3}	Dislocation density (ρ) x 10^{16} (cm^{-2})	Band gap (eV)
0	0.0116	12.88	853	2.81	0.55	1.92
1	0.0169	8.88	847	4.07	1.16	2.00
2	0.0198	7.57	845	4.78	1.59	2.15
3	0.0218	6.87	835	5.27	1.94	2.33
4	0.0233	6.44	833	5.62	2.20	2.44
5	0.0285	5.25	830	6.88	3.31	2.57

3.2. Morphological properties

It is known that additive level plays a crucial role in the deposition process. Additive content not only affects the reaction rate but also the morphology of the fabricated nanostructures. The morphological analysis of the film deposited absence of saccharin and PbS thin films deposited from the solution containing different concentrations of saccharin were examined with SEM images. SEM images of PbS nanoparticles that fabricated absence and presence of the saccharin are shown in Figure 2. It can be seen from the figures that surface change of up to 3% adding level was clearly observed. It is clear that the crystalline size of the samples was decreased with the increase of an amount of saccharin in the bath. With further increase in additive concentration, the surface change appears little differences. On the other hand higher saccharin addition did not have a significant effect on the surface appearance of the lead sulfide (PbS) nanoparticles.

It is obvious that surface roughness of the fabricated films can be affected the presence of the additive in the growth solutions. We have used Arithmetic Mean Roughness (Ra) value to explore the effect of saccharin content on the surface roughness. The results indicated that surface roughness (Ra) of the coatings influenced considerably by the additive rate. Ra values of the PbS chalcogenide films fabricated with or without saccharin were tabled in Table 1. The effect of the additive concentration on the surface roughness of the chalcogenides is also displayed in Figure 3. It can observe from Table 1 that measurement of each sample was done in triplicate and surface roughness parameters are expressed as an average of three determinations and standard deviation value. From Table 1, Ra values of the chalcogenide films are measured as 0.56 (without additive), 0.48 (1% saccharin added), 0.40 (2% saccharin added), 0.36 (3% saccharin added), 0.30 (4% saccharin added) and 0.28 µm (5% saccharin added) respectively. These values show that concentration of saccharin in the growth solutions play an important role in the roughness of PbS coatings.

We have calculated percentage relative standard deviation (%RSD) of the Ra data to the indicate precision of the measurements. It is known that RSD represents the precision and it should be less than 10%. In this work, repeatability of the measurements was found to be satisfactory for the samples with RSD % values between the ranges of 1.79% and 7.06%. Precision did not exceed 10% in any case indicating the acceptable repeatability of the procedure.

3.3. Optical properties

The optical absorption and transmittance spectrum for the films fabricated without and with saccharin addition were studied via spectrophotometric method. E_g values were calculated via Tauc formula [18]. High absorption region was used to calculate band gap by assuming a direct transition between valence and conduction bands. E_g of a direct band gap semiconductor can be obtained from the plot of $(\alpha h\gamma)^2$ versus $h\gamma$ by extrapolating the straight line portion of the absorption edge to $(\alpha h\gamma)=0$. Figs 4 and 6 shows that E_g and transmittance results of all coatings (depending on the different level of saccharin in the growth solutions).

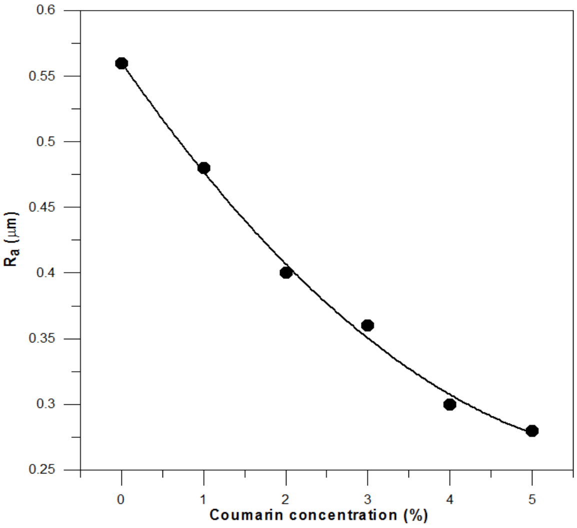

Figure 3. Surface roughness parameters of PbS coatings as a function of Saccharin levels in the baths.

As seen from Figure 4, the band gaps of the films prepared at various additive content were found to be 1.92, 2.00, 2.15, 2.33, 2.44 and 2,57 eV respectively. The results show that the E_g values increase gradually with increasing additive level in the bath solution though the sample are in same range of thickness. The decrease of crystallite size of the films deposited at different saccharin concentrations is believed to be the main reason for the blue shift of optical band gap. It can also be seen from Figure 5 that variation between crystallite size and band gap as a function of saccharin content has reverse behaviour. It was observed that with increasing of the additive level, the crystallite size of the PbS coatings reduced (from 12.88 to 5.25 nm) and E_g value of the nanostructured films increased (from 1.92 to 2.57 eV). This may be related to the effect of quantum confinement. The quantum confinement effect is highly related to the exciton Bohr diameter. Metal chalcogenide semiconductor PbS has a large exciton Bohr diameter (~36 nm) which shows quantum confinement effects. If electron and hole motions are confined in three-dimensional spaces, this phenomenon leads to quantum confinement effect. This effect is observable if the particle size of the nanostructured thin films is comparable or smaller than its Bohr exciton diameter which acts as a natural length scale of the electron-hole pair [2, 12].

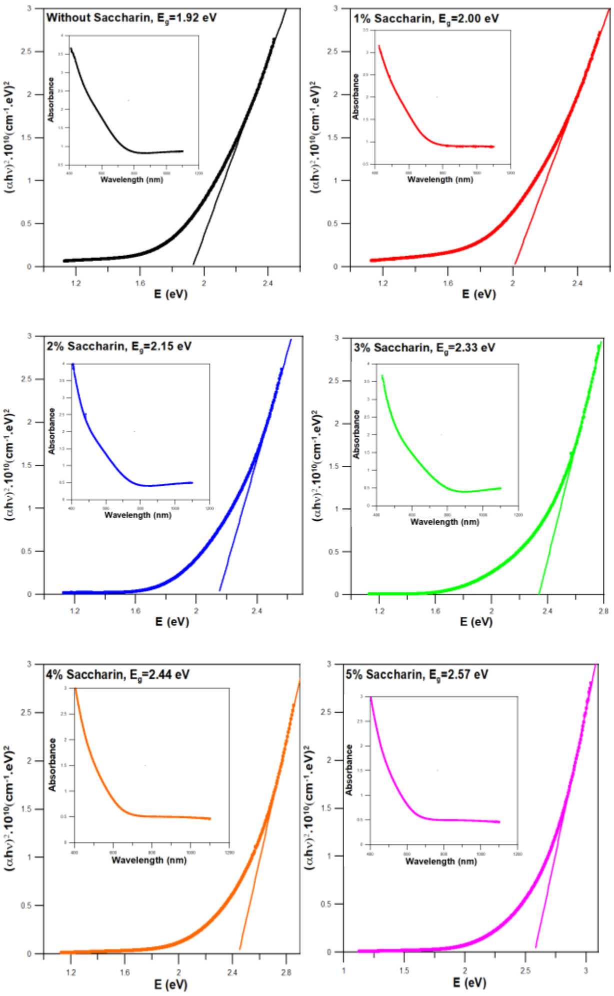

Figure 4. Band gaps of the PbS films according to the different level of saccharin in the growth solutions

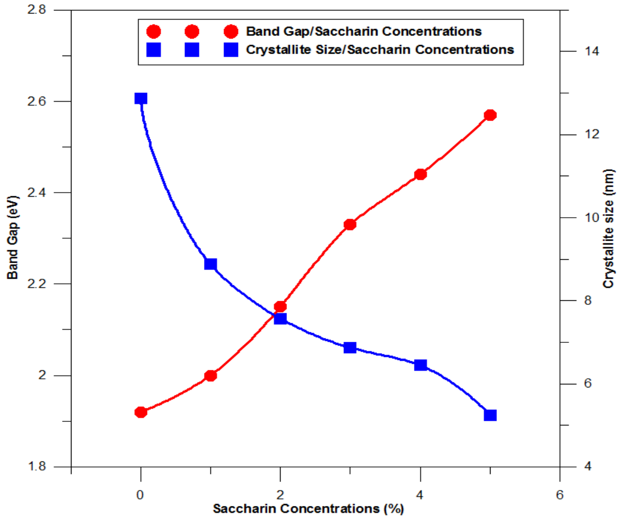

Figure 5. Variation between crystallite size and band gap as a function of Saccharin concentrations in the growth solutions.

It was observed from Figure 6 that the optical film transparency increased with increasing saccharin addition in the growth baths. According to the figure, the transmittance in the wavelength range 650-950 nm of the samples was found to be ~12.9, ~15.0, ~19.2, ~40.4, ~34.6 and ~34.2% as a function of saccharin additive contents respectively. It is clearly suggests that the transmittance was affected by saccharin level added in the growth solution. This increase in optical transmittance may be related to changing of the surface properties.

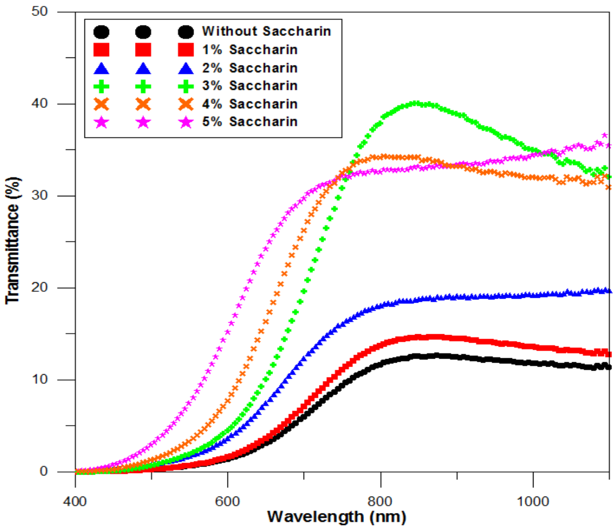

Figure 6. Optical film transparency depending on saccharin level in the baths.

Furthermore, it is clearly seen from the optical transmittance spectra that the absorption edge is between 420 nm and 620 nm for all films. Additionally, it can be observed that the absorption edge shifts towards shorter wavelength side as the saccharin concentration increases. This blue shift of the absorption edge indicates a decrease of the crystallite sizes and increase of optical band gap of the samples also presence of quantum confinement effect [19].

4. Discussion and Conclusion

Lead sulfide (PbS) chalcogenide films were synthesized as a function of saccharin content by chemical bath deposition. The influences of the additive on surface roughness, surface morphology, optical and structural properties of the coated films were investigated. The results observed that addition of saccharin in the growth process plays an important role in the structural, morphological and optical properties of the films. Crystallite sizes of the nanostructures were found to be in the range of 12.88-5.25 nm. The optical absorption result showed that E_g value of PbS thin films increased with increasing saccharin content. Additionally, obtained optical transmittance results indicated that saccharin added chalcogenide PbS coatings are more transparent than the PbS nanostructures without saccharin. Blue shift in the absorption edge and widening of the band gap of PbS thin films were achieved by coumarin addition. Morphological results displayed that surface roughness value of the PbS films seems to depend on the saccharin addition. The surface roughness of chalcogenides decreased substantially depending on additive level. Finally, these results indicate that saccharin addition in the growth baths is an important factor for the morphological, structural and optical properties of coated chalcogenide films and due to its good optical properties saccharin added PbS thin films may be suitable for use as the buffer layer of thin film solar cells.

Acknowledgment

The authors thank to the Scientific Research Coordination Unit of Mustafa Kemal University for providing financial support to the project (16042).

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