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## **ЛЮМИНЕСЦЕНЦИЯ СОЕДИНЕНИЙ ПОЛИВИНИЛОВЫЙ СПИРТ – CdS С ДОБАВКАМИ Cu, Al И Mn**

Синтезированы соединения поливиниловый спирт – CdS с добавками Cu, Al и Mn. Предполагается, что эти соединения обладают одномерной (полимерной) структурой. Их спектры люминесценции исследовались с целью установления структуры путём сравнения с люминесцентными свойствами других материалов на основе CdS. Установлено, что добавка меди приводит к тушению люминесценции, тогда как интенсивность люминесценции соединения с добавкой марганца достигает максимума при определённой его концентрации, а добавка алюминия не оказывает на неё существенного влияния. Вид спектров люминесценции при допировании существенно не изменяется. Эти спектры, однако, существенно отличаются от спектра кристаллического CdS и похожи на спектры CdS, растворённого в стекле. Также было обнаружено, что люминесценция соединений сильно поляризована, люминесценция стёкол поляризована в меньшей степени, а люминесценция кристаллического CdS не поляризована.

**Ключевые слова:** нанокompозиты, CdS, люминесценция, структура CdS, неорганические полимеры.

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## **LUMINESCENCE OF POLYVINYL ALCOHOL – CdS COMPOUNDS DOPED WITH Cu, Al AND Mn**

Polyvinyl alcohol – CdS compounds doped with different concentrations of Cu, Al and Mn were produced. It is expected that these compounds have one-dimensional (polymeric) structure. Their luminescence spectra were investigated with the aim of use of luminescence for the structure characterization through comparison of luminescent properties of compounds and other CdS based materials. It was found that addition of copper leads to luminescence quenching while intensity of luminescence of compound doped with manganese reaches maximum at some concentration of additive and aluminum does not influence noticeably on the luminescence intensity of the compound. Luminescence spectra were not significantly changed at the doping. They are different from spectrum of crystalline CdS and similar to luminescence of CdS dissolved in glass. It was also found that luminescence of compounds is highly polarized, luminescence of glass is less polarized and luminescence of crystal CdS is not polarized.

**Keywords:** nanocomposites, CdS luminescence, CdS structure, inorganic polymers.

## **Introduction**

Semiconductors have attracted big attention due to their optical properties as well as their application in the optoelectronic technology. Doped semiconductors have caught special attention due to their advantages. Semiconductor luminophors compose an important class of luminescent materials. Cadmium sulfide is an important representative of this class and compounds of the second and six group elements ( $A^2B^6$ ) of the periodic table [1]. In this research we investigated luminescent properties of polyvinyl alcohol (PVA) – cadmium sulfide compounds doped with copper, aluminum and manganese. It is known that doping of  $A^2B^6$  compounds with copper, aluminum and manganese leads to change of conductivity [2,3] and appearance of activated luminescence [1,4-14]. We synthesized cadmium sulfide doped with the mentioned elements as a part of polymeric compound. It is commonly accepted that CdS forms 3-dimensional particles (or quasi 0-dimensional quantum dots) in polymeric matrices [15-20] and glasses [7,8]. PVA is known as complexing polymer due to presence of large amount of hydroxylic groups in its structure [21]. It was suggested that CdS can form one-dimensional polymeric chain like:  $-(Cd-S)_n-$  in PVA matrix [22]. Existence of CdS based coordination polymers were also mentioned in literature [19]. Such one-dimensional structures are objects of special interest due to their possible unusual properties like high-temperature superconductivity [23]. On the other hand, it is difficult to prove the forming of uncommon for CdS non-crystalline one-dimensional structure. Analysis of the differences of crystalline and non-crystalline specimens' properties can be used for this purpose. For example, reported luminescence of polymers is highly polarized [24-27]. It is difficult to expect such degree of crystal phosphors' luminescence polarization due to migration of exited charge carriers [1]. Therefore, the observation of polarized luminescence can give confirmation of polymeric structure. Another peculiarity of luminescent properties of one-dimensional structures is expected dependence of luminescence intensity from dopant concentration. Commonly, the optimal concentration of activator exists, at which luminescence intensity reaches its maximum. It can be explained by interaction of neighbor activators which can lead to concentration quenching of luminescence. We suppose that this optimal concentration should be higher for one-dimensional system in comparison with three-dimensional one because additional dimensions give more possibilities for activator atoms' interaction. Structure of luminescent centers

also can be different for one- and three-dimensional systems, what can lead to difference of their luminescent properties. The aim of this research is to check these suppositions. Simultaneously, we observed influence of common dopants on the luminescence of PVA-CdS compounds with the aim to estimate prospectiveness of their usage in optoelectronics. Chosen dopants Cu(II) and Mn(II) are typical isovalent impurities in  $A^2B^6$  compounds. And Al is typical heterovalent impurity (for  $A^2B^6$ ) which promotes intrinsic defects formation. It seems unlikely that in one-dimensional system it acts similar to three-dimensional case – producing cation vacancies because it should terminate polymeric chains:  $S=Al-(S-Cd)_n$ .

## **1. Experimental procedure**

### **1.1. Chemicals**

We used PVA 18-88 grade, produced by BDH Chemicals Ltd.,  $CdCl_2 \cdot 2.5H_2O$  (the main additive) “analytically pure” grade with provided Cu impurity  $5 \cdot 10^{-4}$  molar ratio. Doping compounds were  $CuCl_2 \cdot 2H_2O$  “analytically pure”,  $AlCl_3 \cdot 6H_2O$  “analytically pure”, and  $MnCl_2 \cdot 4H_2O$  “pure”. Sulfurization was carried out with  $H_2S$  produced from  $Na_2S \cdot 9H_2O$  “analytically pure” and HCl “pure” interaction. For comparison we used microcrystalline CdS powder “chemically pure” grade.

### **1.2. Sample preparation**

We prepared series of films with the nearly same sizes and different concentrations of the dopants. Firstly we produced 10 % water solution of PVA. Then, we weighted 0.02 g of additive ( $CuCl_2 \cdot 2H_2O$ ,  $AlCl_3 \cdot 6H_2O$ , or  $MnCl_2 \cdot 4H_2O$ ) and dissolved it in 20 ml distilled water. 0.65g of  $CdCl_2 \cdot 2.5H_2O$  was added to 8 ml, 4 ml, 2 ml, 1 ml of such solutions and diluted by water to 10 ml. This amount was mixed with 5 ml of 10 % PVA solution. This mixture was spilled and left on a flat fluoroplastic surface until completely dryness of the sample at room conditions. After the samples become dry they were placed in glass vessel with  $H_2S$  atmosphere for several days to convert halides into sulfides. After sulfurization and drying the samples were ready for luminescence measurement. Finally, we expect that dopants are included homogeneously in CdS and make nearly  $10^{-3}$ - $10^{-2}$  molar ratio of CdS. There were no crystalline phases in the specimens according to X-ray analysis. It should be also mentioned that intention to amorphization of small CdS nanoparticles was reported earlier [28]. For comparison we used the reference sample which was produced by mixing of PVA solution with CdS microcrystalline powder

with the components ratio 5 ml of 10 % PVA to 0.2 g CdS. This sample was opaque in opposite to other transparent samples. As other reference sample we also used standard CdS based color glass.

### **1.3. Luminescence measurements**

Luminescence measurements were carried out at room temperature using spectrometric complex produced by “OKB Spectr” company, Russia. It is not serial device. It was assembled from standard units on individual request. In our research we additionally fitted this device. Luminescence was immediately excited by full UV LED radiation with wavelength  $\lambda_{\max} \sim 405$  nm. UV LED was supplied by stabilized current (10 mA). Luminescent radiation was preliminary separated by standard CdS based color glass (GS-12), passed through grate monochromator “MDR-41” and registered with photomultiplier. The spectra were not corrected. Series of spectra of doped specimens were recorded at similar conditions (slits' width, LED current, photomultiplier voltage etc.) to allow luminescence intensity comparison. Spectral resolution:  $\sim 5$  nm. Reference spectrum of PVA-CdS mixture (Fig. 1) was recorded at wider slits' width and higher LED current due to signal weakness. Correction factor (300) was estimated and used for the data comparison.

## **2. Results and discussion**

It was observed that standard CdS based color glass used for exciting radiation reduction also gives weak yellow luminescence similar to luminescence of PVA-CdS compounds. On the one hand it gives undesirable influence on the results. But on the other hand this luminescence can give valuable information about dependence of CdS luminescence from its structure. Therefore, we used it as reference sample. Luminescence spectra of PVA-CdS compound, CdS based standard color glass and PVA-CdS mixture (multiplied by correction factor) are represented in fig. 1. There we can see two bands. The band at 450 nm is a “tail” of exciting radiation. We don't exclude it because it gives information about excitation/emission rate. The band with maximums at 530 or 540 nm we attribute to luminescence of CdS dissolved in glass and to PVA-CdS compound correspondingly. There is expected decrease of excitation band intensity after passing through the sample and color glass in comparison with passing only through color glass. The maximum of yellow band luminescence in PVA-CdS mixture (reference sample) is observed nearly at 570 nm. In our opinion, a sufficient difference in luminescent properties reflects differences of CdS structures. Similar decrease of luminescence maximum wavelength with decrease of CdS particle size was earlier reported [29].

This observation is in concordance with our results. We also suppose that luminescence of color glass does not influence appreciable on the results for PVA-CdS compound luminescence due to its weakness.

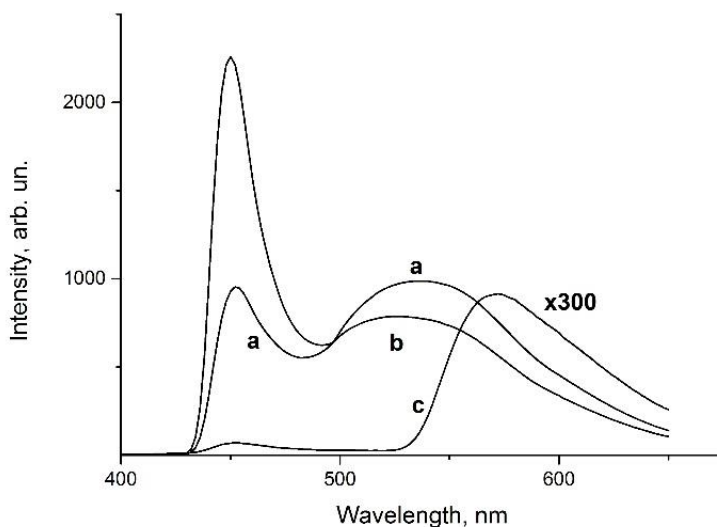


Fig. 1. Luminescence spectra of combination of PVA-CdS compound specimen and CdS based color glass (a), CdS based color glass without specimen (b) and PVA-crystalline CdS mixture (with correction factor 300) (c)

Luminescence spectra of pristine PVA-CdS compound and compounds doped with Cu are represented in fig. 2. The intensity of luminescence permanently decreases with increase of Cu content. The position of luminescence band weakly changes with varying of Cu concentration. Based on these data we can conclude that Cu rather quenches luminescence of CdS in PVA-CdS compound. Another explanation can be given taking into account absorption of the samples. The samples with higher content of Cu look more blackish. It means that decrease of luminescence intensity can occur due to higher absorption of exiting and luminescent radiation in specimens with higher Cu content.

Similar decrease of CdS nanoparticles luminescence intensity at Cu doping was observed in other research [4] but luminescence spectra there were significantly different from ours ones. They consisted of two bands at 510 and 530 nm. Dopant concentrations were sufficiently higher then in our

research. As a result we can conclude that our specimens have other structure and properties then known crystalline CdS luminophors and nanoparticles doped with Cu.

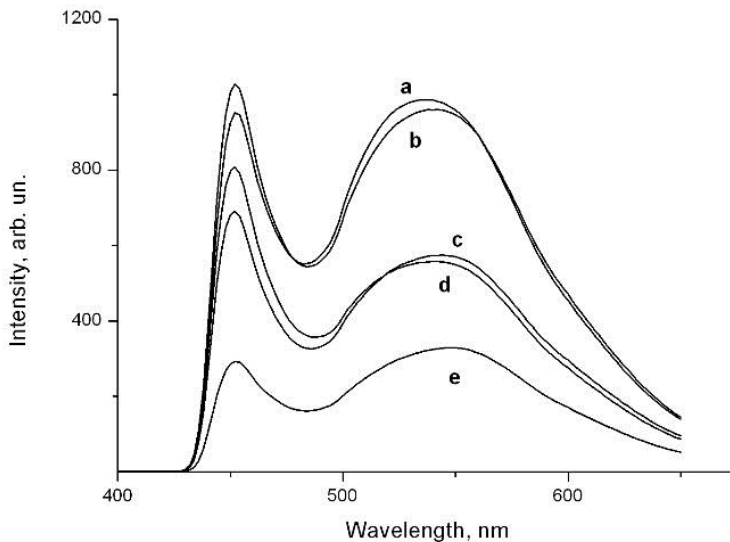


Fig. 2. Luminescence spectra of PVA-CdS compounds without doping (a) and doped with copper at molar ratio  $2 \cdot 10^{-3}$  (b),  $4 \cdot 10^{-3}$  (c),  $8 \cdot 10^{-3}$  (d) and  $1,6 \cdot 10^{-2}$  (e)

Luminescence spectra of pristine PVA-CdS compound and compounds doped with Al are represented in fig. 3. There we can see the same two bands. The positions of luminescence band weakly change with varying of Al concentration. Intensity of CdS luminescence changes insufficiently at dopant concentration lower then  $3 \cdot 10^{-3}$  molar molar ratio and decreases at higher concentrations. Intensity of blue band drops more rapidly. Some inconsistency in its changing can be attributed to experimental inaccuracy. It seems that addition of Al leads to increase of absorption in UV region. Permanent slight long-wave shift of luminescence maximum is also observed at Al concentration increase.

We did not find data about activation of CdS luminescence by doping with Al in literature. But there is report about activation of CdS luminescence by doping with In and Cl [30] which should have the same mechanism as at Al doping. In this report significant change of luminescence band position within 480-600 nm regions was observed. We observe luminescence band in the same region but with different impurity concentration dependence. It can

mean that doping of our system with three-valence cations entails other structural consequences than for bulk CdS.

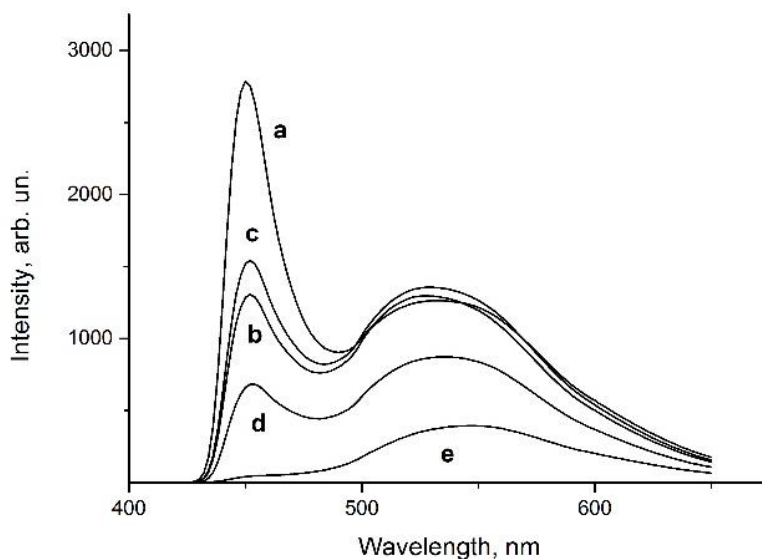


Fig 3. Luminescence spectra of PVA-CdS compounds without doping (a) and doped with aluminum at molar ratio  $1,4 \cdot 10^{-3}$  (b),  $2,8 \cdot 10^{-3}$  (c),  $5,6 \cdot 10^{-3}$  (d) and  $1,1 \cdot 10^{-2}$  (e)

Concentration dependence of luminescence of PVA-CdS compounds doped with manganese is represented in fig. 4. It is different from that of specimens doped with copper and aluminum. Intensity of CdS luminescence changes unconformable to varying of dopant concentration. It reaches maximum at Mn molar ratio nearly  $5 \cdot 10^{-3}$ . It seems that manganese addition can temperately enhance luminescence of PVA-CdS without significant change of luminescence band position.

There are lots of researches of luminescent properties of crystalline CdS particles doped with Mn but they report quite controversial results. CdS (pristine and Mn-doped) luminescence bands positions were reported in 450-500 [5,6,10], 500-600 [6-8,12-14] and 600-800 [5-9,11] nm regions. At Mn concentration nearly 1-10% increase [6,12,14] or decrease [5] of CdS particles luminescence intensity were reported. This used doping level is much higher than usual activator concentration in crystalline CdS luminophors. Thus, it is difficult to use results of other researches for comparison purposes. Possibly, Mn doping “activates” luminescence of PVA-CdS compounds to some extent and it needs in further investigation.

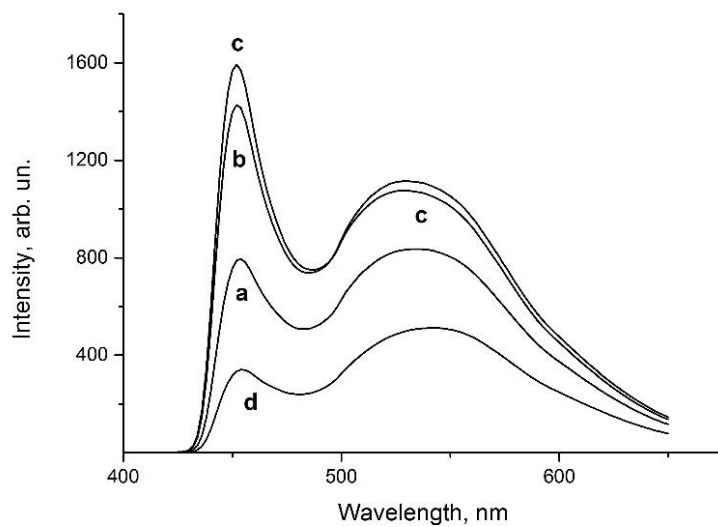


Fig 4. Luminescence spectra of PVA-CdS compounds doped with manganese at molar ratio  $1,7 \cdot 10^{-3}$  (a),  $3,4 \cdot 10^{-3}$  (b),  $6,8 \cdot 10^{-3}$  (c) and  $1,3 \cdot 10^{-2}$  (d)

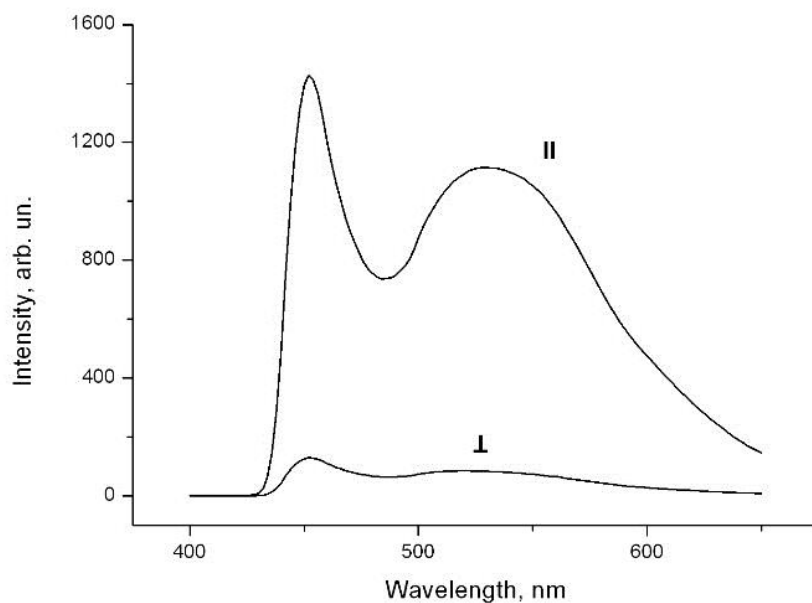


Fig. 5. Luminescence spectra of PVA-CdS compound doped with Mn at  $3,4 \cdot 10^{-3}$  molar ratio at polarizers in parallel and perpendicular positions



## Conclusion

Polarized luminescence spectra of PVA-CdS compound doped with Mn ( $3,4 \cdot 10^{-3}$  molar ratio) are represented in fig. 5. They indicate that luminescence of PVA-CdS compound is highly polarized. The degree of polarization can be expressed by the ratio of luminescence intensities at parallel and perpendicular polarizer's positions:  $I_{\parallel}/I_{\perp}$ . For the estimation of polarization degree we subtracted baseline between wavelength  $\lambda=480$  nm and  $\lambda=650$  nm and used intensity value at maximum of luminescence band.  $I_{\parallel}/I_{\perp}=16.7$  was found for the spectra from fig. 5, 5.7 for the CdS colored glass and nearly 1 for PVA-microcrystalline CdS reference sample. These results confirm polymeric structure of produced PVA-CdS compounds.

It was observed that doping of PVA-CdS compounds with Cu, Al and Mn can differently effect on intensity if its luminescence. Impurity of Al evidently has no sufficient influence on PVA-CdS compound luminescence efficiency to some extent and slightly increases luminescence maximum wavelength. The activation of this compound luminescence by Cu addition also was not observed. Mn doping seemingly can moderately enhance luminescence at molar ratio nearly  $5 \cdot 10^{-3}$ . This value is quite higher than that for common crystalline doped CdS luminophors. It was also found that yellow luminescence band shifts significantly to shorter wavelength at PVA-CdS compound in comparison with crystalline CdS and becomes similar to the luminescence of CdS doped glass. High degree of PVA-CdS compounds' luminescence polarization was detected. These observations can be attributed to the difference in CdS structures, at least to decrease of particle size.

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