Characterization of PbS nanoparticles trapped in Y zeolite by X ray diffraction and scanning electron microscopy

H. Tebani , N. Keghouche
Laboratoire Microstructure et Défauts dans les Matériaux, Université Frères Mentouri-Constantine1, Route de Ain El Bey, 25110 Constantine, Algeria
tebani.houda@gmail.com

Abstract
The present study examines the PbS semiconductor nanoparticles trapped in Y zeolite synthesized at room temperature via a simple chemical reaction when 2-mercaptoethanol noted RSH was used with a different amounts as a sulfur source. The obtained samples were calcined at various temperatures (T = 100 - 550°C). X ray diffraction (XRD) and scanning electron microscopy (SEM) were used to characterize the samples at different steps of their elaboration. After thiol addition, the X-rays diffraction reveals the formation of galena lead sulfide phase. The PbS crystallite size, is about 20 nm. After calcinations XRD patterns present the features of PbS. These results are in good agreement with SEM observations which show rod-shapped crystallites corresponding to the first series of samples (ϕ = 20-50 nm, l = 5-10 μm). In the second series the crystallites form clusters at 550°C with an average size of 20-50 nm.

Keywords: Y zeolite; semi-conductor; nanorods; PbS; thiol; clusters.

Introduction
Nanoscale materials take attention of many scientists because of their special properties [1], different from that of the bulk state. Particularly, semi-conductor quantum dots present size dependant optical properties [2-3-4], which give them a choice place in several applications such as photonics and medical imaging.

Experimental
Semiconductor nanoparticules were synthesized by the chemical reaction at room temperature of a solution containing PbY and thiol which plays the role of sulfur atom source, the solvent used in all experiments was water of ultra-high purity. The chemicals were of highest quality and used as received: from Biochem for sodium silicate (NaO2Si), sodium hydroxide (NaOH) and lead acetate (Pb2(C2H4O3)2), Prolabo for ammonium hydroxide (NH4OH), Aldrich for 2-mercaptoethanol (HOCH2CH2SH), sodium aluminate (AlO2Na) was synthesized. In a first step, we synthesized NaY faujasite by the sol-gel route [5]. In a second step, the lead ions were fixed on the zeolite by ionic exchange. Finally, a various amount of thiol was added to the obtained samples, named PbY and calcined at various temperatures (T = 100 - 550°C).

Results and discussion
The XRD study reveals a better cristallinity of PbY than NaY. After the thiol addition, galena lead sulfide phase cubic centered faces structure was detected. Varying the amount of thiol from 10-3 to 10-2 M, the PbS crystallite size, estimated from Scherrer equation, is about 20 nm. At this stage, the nanoparticles couldn't be observed by SEM. After calcinations XRD patterns present the features of PbS.

These results are in good agreement with SEM observations which show rod-shapped crystallites corresponding to the first series of samples R= [RSH]/[Pb+2] =1 (ϕ = 20-50 nm, l = 5-10 μm) and in the second one R=[RSH]/[Pb+2] = 10 clusters were observed at 550°C with an average size of 20-50 nm.

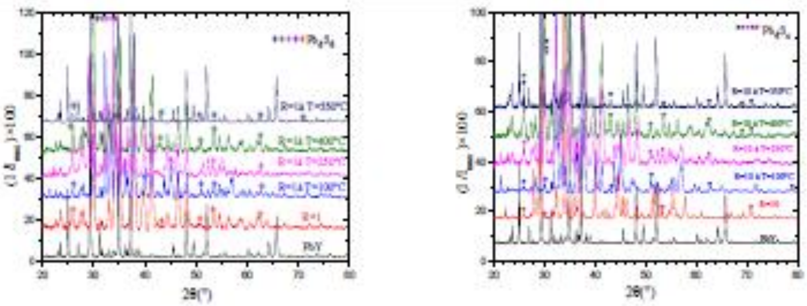

Figure 4: XRD patterns of PbS nanoparticles at deffrent calcination temperatures: (a) Samples containimg 10^{-3}M of RSH, (b) Samples containimg10^{-2}M of RSH.

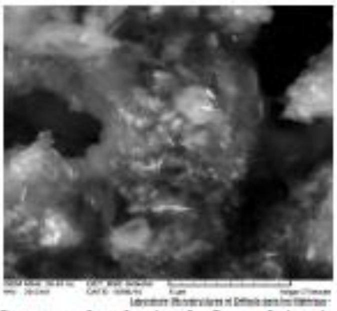

Figure 2: SEM image of: (a) PbS nanorods, obtained after calcination at 550°C, the sample containing 10^{-3}M of RSH, (b) PbS Clusters, obtained after calcination at 550°C, the sample containing 10^{-2}M of RSH .

Table 2.Structural parameters and particle size of PbS phase.

The samples		T (°C)	Phases	Cell paramrters (nm)	Structure	ϕ (nm)
$R = [RSH]/[Pb^{+2}] = 1$	Before calcination	-	Pb_4S_4	a = 0,5936	FCC	21
	After calcination	100		a = 0,5934		26
		250		a = 0,5938		30
		400		a = 0,5940		21
		550		a = 0,5940		41
$R = [RSH]/[Pb^{+2}] = 10$	Before calcination	-	Pb_4S_4	a = 0,5944	FCC	47
	After calcination	100		a = 0,5939		35
		250		a = 0,5933		23
		400		a = 0,5940		20
		550		a = 0,5934		50

Conclusion

The structural study reveals the formation of lead sulfide phase. Varying synthesis conditions crystallites of different size were obtained, after calcination PbS nano-rods and clusters were formed at 550°C

observed by SEM. This type of composite has a choice place in several applications such as photonics and medical imaging due to their properties.

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