

ELABORATION AND CHARACTERIZATION OF HYBRID MATERIALS ORGANIC / INORGANIC

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Résumé

Les matériaux hybrides "organique-inorganique" font l'objet d'un immense intérêt, permettant à la fois de combiner certaines propriétés d'un matériau inorganique et d'un polymère. Dans ce travail nous avons réalisé une étude sur les polymères conducteurs, en général, mettant l'accent sur la polyaniline. D'autre part, nous avons présenté les composés inorganiques ($NbSe_2$, Nb_3Sn et le composé ternaire $SnNb_5Se_9$). A partir de la méthode chimique, nous avons pu synthétiser les mélanges suivants: PANI/ $NbSe_2$, PANI/ Nb_3Sn et PANI/ $SnNb_5Se_9$. L'étude structurale de ces nouveaux composés a été réalisée par la diffraction des rayons X et la spectroscopie infrarouge. La morphologie des mélanges obtenus a été réalisée par microscopie électronique à balayage.

Mots-clés: hybride, la polyaniline, polymérisation, Structure.

Abstract

Hybrid materials "organic-inorganic" are the subject of immense interest, allowing both to combine some properties of an inorganic material and a polymer. In this work we have carried out a study on conductive polymers, in general, emphasizing the polyaniline. On the other hand, we have presented the inorganic compounds ($NbSe_2$, and ternary compound Nb_3Sn $SnNb_5Se_9$). From the chemical method, we had to synthesize the following mixtures: PANI/ $NbSe_2$, PANI / Nb_3Sn PANI / $SnNb_5Se_9$. The structural study of these new compounds are produced by X-ray diffraction and infrared. The morphology of the resulting mixtures to be studied by scanning electron microscopy.

keywords: Hybrid, Polyaniline, Polymerization, Structure.

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ملخص

المواد الهجينة المتكونة من المواد العضوية و الغير العضوية اصبحت في الاونة الاخيرة ذات اهمية بالغة و هي بدورها تسمح بدمج خصائص مادة غير عضوية والبوليمر. خلال هذا العمل، أجرينا دراسة عن البوليمرات الناقلة، بصفة عامة ، وخصوصا البوليانيلين. من ناحية أخرى، قدمنا المركبات غير العضوية $PANI/Nb_3Sn$ و $PANI/NbSe_2$, $PANI/SnNb_5Se_9$. ويتم دراسة الهيكلية لهذه المركبات الجديدة من قبل XRD و الطيفي بالأشعة تحت الحمراء. وقد تمت دراسة مورفولوجية المخاليط الناجمة عنها عن طريق فحص المجهر الإلكتروني

الكلمات المفتاحية: الهجين، بولي أنلين، البلمرة، الهيكل .

I

ntroduction

A hybrid material is a system in which coexist both organic inorganic species. Some hybrids are the most representative and are derived from industry hinges and polymers, where inorganic pigments or fillers are dispersed in organic components (solvents, surfactants, polymers) to improve the optical and mechanical properties. But the concept of organic-inorganic hybrid materials appeared only very recently with the birth of green chemistry [1] shifted research towards more sophisticated and nanocomposite structures with original value larger. It is within this framework that fits the synthesis of organic inorganic hybrid materials. Have examples include the use of hybrid materials in areas such as opto-electronics [2], catalytic systems[3], or domain medical pharmaceutique [4].

The polyaniline is a polymer composed of amine and imine units is mainly studied and used for his qualities of electronics driver. This polymer can be prepared by two methods, one chemical [5-6] and other electrochemical[7-8]. the most preferred method for synthesis of PANI composite is to use either HCl H₂SO₄ with ammonium peroxydisulfate as an oxidant. or Each involves the oxidation of aniline in acidic media. The polyaniline emeraldine salt (PANI-ES) was synthesized by oxidation of aniline with ammonium peroxydisulfate in a weak hydrochloric acid [9].

By definition, the compounds MX₂ up by stacking successive sheets that are at arrangement of atomic layers, the formula contains from: M transition metal element of Group II, III, IV and X and VB chalcogen (selenium or sulfur). In this work, we are interested primarily in VB diselined group metal (Nb). Each sheet of this stack of two-dimensional lamellar structure consists of trios atomic layers, two layers of chalcogen (X) in the central metal in a few words it takes the form XMX like a sandwich, but connections between the stack of sheets low-type van der Waals interactions in each sheet strong.

Since the discovery of superconducting A-15, in 1954, thousands of original publications appeared on this subject [10,11]. The true history of these so-called high-temperature phases criticism began with the synthesis of V₃Si (T_c = 17K) and Nb₃Sn (T_c = 18.3K) [12]. Inclusion compound Nb₃Sn channels in 2H-polytype NbSe₂ trigonal prismatic structure. Thus is. achieved the synthesis of a new derivative[13]. The mixture is made from 5% or 6% of Nb₃Sn purity (99.9%) in NbSe₂, the reaction favors transport [processes such as iodine preparation NbSe₂, in this case we use tin in the gaseous state.

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2.Experimental

From the method of polymerization, we

synthesized PANI/NbSe₂ the mixture, and the compound PANI/Nb₃Sn SnNb₅Se₉. Previously, 5ml of aniline was dissolved in hydrochloric acid bidistilled molarity of 1M. The mixture is introduced into a 250 ml flask, then added directly to 5% (NbSe₂, Nb₃Sn and SnNb₅Se₉). The two starting materials have been mixed by stirring for 1 h at 4 ° C. A second solution of ammonium persulfate, prepared beforehand, was added dropwise over 30 min keeping the reaction mixture with constant stirring to the initial temperature. Stirring continues for four hours until the mixture has turned into powdered solid yellow and green. The crude product was recovered by filtration and washed with 100 ml aliquot (50 v / v) methanol / water to remove oligomers may be present.

3. Result and discuss

3.1. Structural Study

After the three X-ray diffraction spectra, we obtained the results of lattice parameter programmatically dicvol04 [14], again produced orthorhombic. Before we search the data banks JCPDS and ICSD has found no results on the resulting compounds, which enabled new products result. The determination of lattice parameters of the diffraction pattern of powder by the program that is based on an input file containing line positions 2θ from stripes and a fitting program which comes with an index file output by giving the results of lattice parameter and the indices of the lines (hkl) leads to the orthorhombic system, with the following parameters:

PANI/NbSe ₂	PANI/Nb ₃ Sn	PANI/ SnNb ₅ Se ₉
a= 7.01359 Å°	a=12.67472Å°	a= 7.01359 Å°
b= 3.40007Å°	b=4.70193 Å°	b= 3.40007Å°
c = 2.50406Å°	c =3.22349Å°	c = 2.50406Å°
	V= 192.11Å° ³	V= 59.71Å° ³

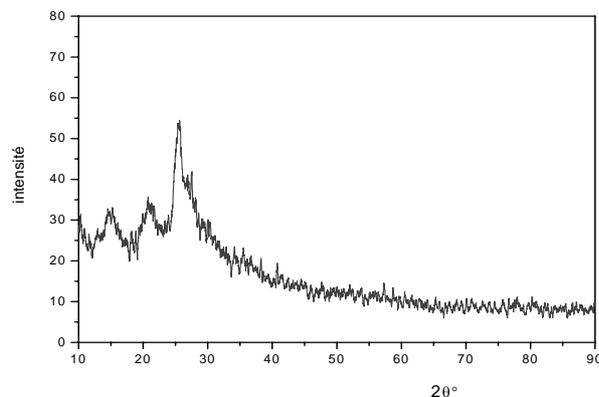


Fig 1: X-ray diffraction spectrum of pure PANI

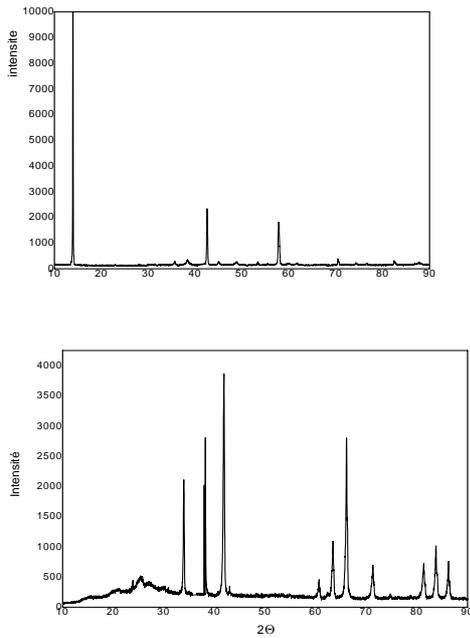


Fig 2: Spectrum of X-ray diffraction of compound NbSe2 and mixing PANI / NbSe2

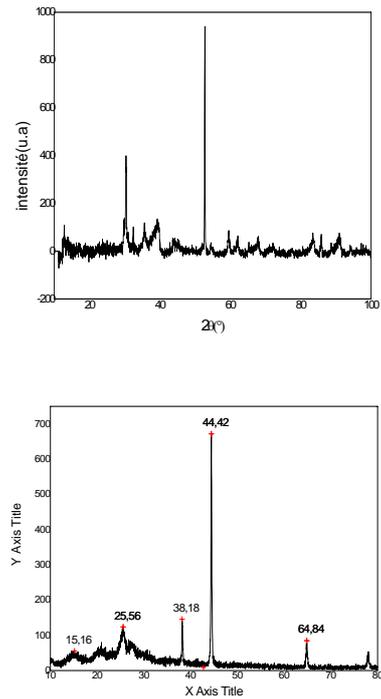


Fig 4: Spectrum of X-ray diffraction of compound Sn Nb₅Se₉ and mixing PANI /Sn Nb₅Se₉

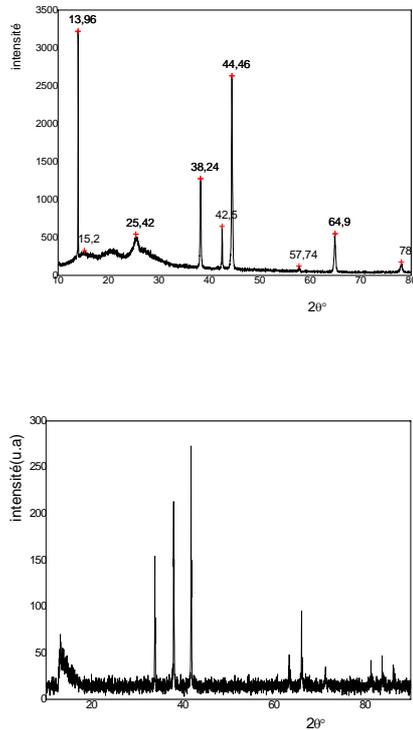


Fig 3: Spectrum of X-ray diffraction of compound Nb₃Sn and the mixture PANI/Nb₃Sn

3.2.The grain size

In particular, if we determine the grain size mixtures PANI/NbSe₂, PANI/Nb₃Sn and PANI / SnNb₅Se₉ using Scherrer law $d = \frac{0.94 \lambda}{\Delta 2\theta \cos \theta}$ [15], with: constant varies between 0.89-1.0, width at half height and position of the line considered, are shown in Table 11, 2 and 3: Represents the size of the grain mixture PANI / NbSe₂, PANI/Nb₃Sn and PANI / SnNb₅Se₉.

Table 1

Pic	couche	centre	mi_hauteur	d(nm)
1	6.98	20.56	1.68	3.62
1	6.83	25.43	1.19	7.13
1	17.36	33.85	0.11	72.75
1	28.20	37.99	0.14	60.72
1	30.37	41.75	0.16	54.63
1	15.80	44.42	0.24	36.06
1	2.70	60.55	0.14	67.07
1	2.87	62.27	0.11	83.64
1	2.70	63.30	0.18	53.99
1	7.54	64.86	0.56	17.34
1	15.16	65.98	0.16	60.02

Table 2

pic	couche	centre	mi_hauteur	d(nm)
1	216.71	13.968	0.05	21.3127
1	362.92	20.572	2.85	2.954
1	190.24	25.507	0.84	10.045
1	253.7	38.243	0.18	46.724
1	72.509	42.518	0.11	78.098
1	651.54	44.465	0.21	41.285
1	16.705	54.78	0.22	41.592
1	148.27	64.867	0.26	37.192
1	38.986	78.013	0.33	31.465
1	34.723	78.013	0.31	34.039

Table 3

pic	couche	centre	mi_hauteur	d(nm)
1	58.68	15.05	2.32	0.47
1	160.44	20.76	2.86	2.94
1	64.931	25.52	0.97	8.73
1	27.62	38.20	0.19	44.57
1	160.77	44.43	0.21	41.11
1	35.36	64.83	0.42	23.33
1	13.83	77.96	0.34	31.16

After the results of new materials, we note that the grain sizes and ranged from (3.62et 83.64nm for PANI/NbSe₂, and 78.09nm 2.95 and 4.7 and 44.57) PANI/Nb₃Sn successive blends and

3.3.Morphology of the resulting compounds SEM

The morphology study is performed by scanning electron microscope (SEM), pure PANI Figure 4 shows that the surface shape is formed by flattened grains of 10µm size is homogeneous, is confirmed by X-ray diffraction spectrum and observed in small particular of de PANI.

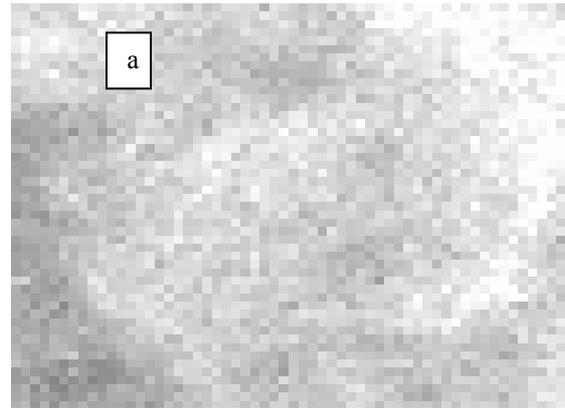
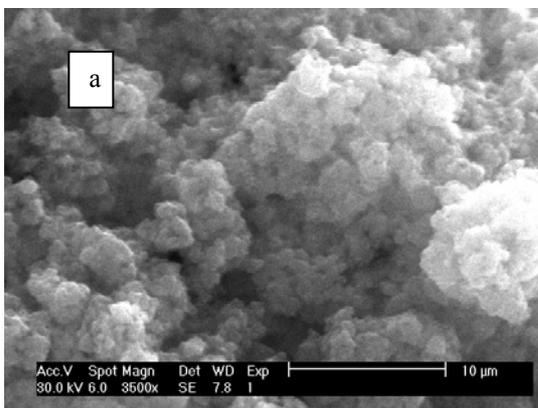


Fig 4 : photon the SEM of PANI

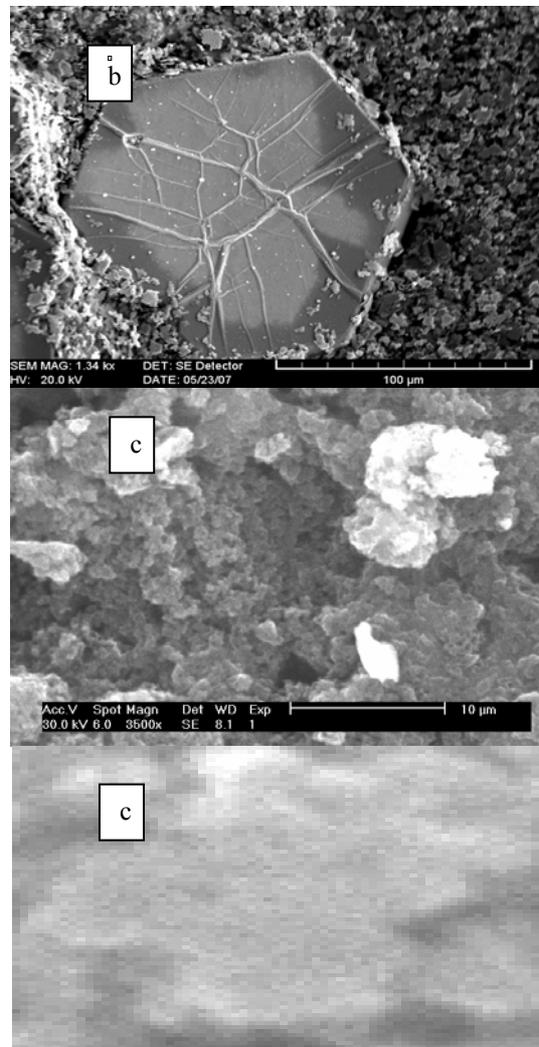


Fig 5 : Scanning electron micrograph of NbSe₂ and PANI/NbSe₂

The figure shows a sample image of the phase which $NbSe_2$ taken SEM It shows consistency in this phase by a large insert size of $20\mu m$ per hex. For mixing PANI / $NbSe_2$ is clear and homogeneous, the $NbSe_2$ disperse in the organic compound as explained in Figure (5c).

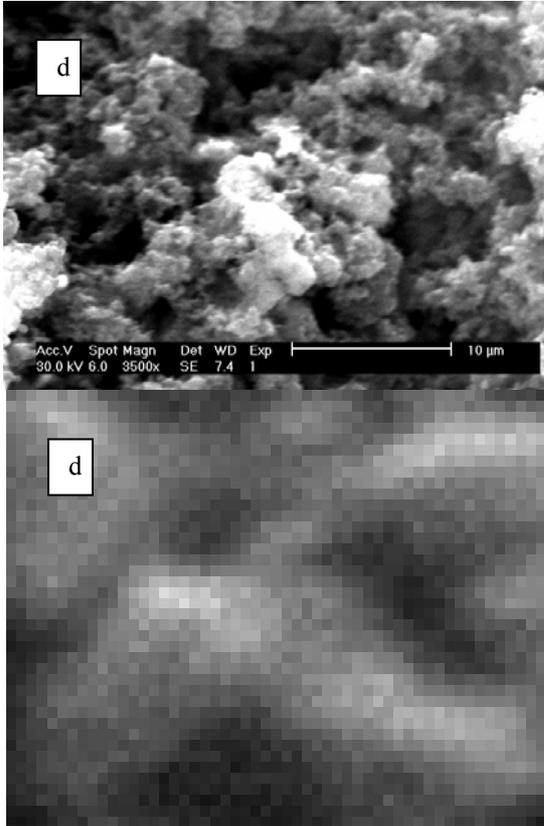


Fig 6 : represented photos of PANI/ Nb_3Sn

The photons of the figure (6e) and (7f) is presented sample PANI/ Nb_3Sn and PANI / $SnNb_5Se_9$, have clear and consistent, but in both pictures there is a surface with holes. They show the latter form of spherical particles would lodge itself in the nuclei of benzene rings to form, at the end a homogeneous hybrid material. Photon $SnNb_5Se_9$ that represents a large hexagonal plate.

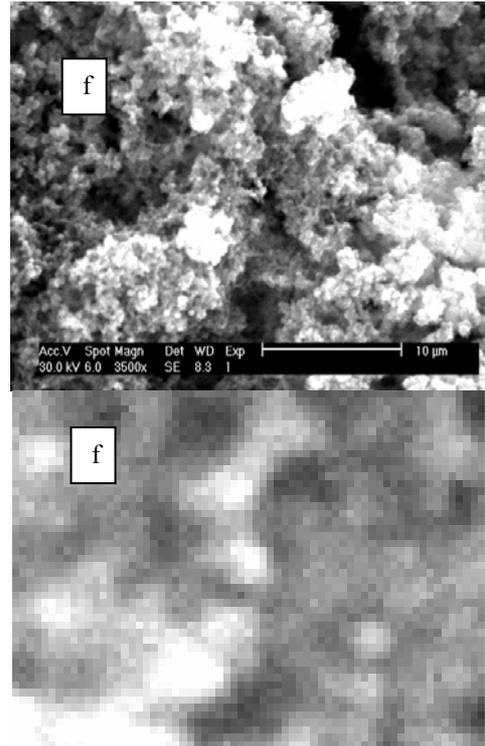
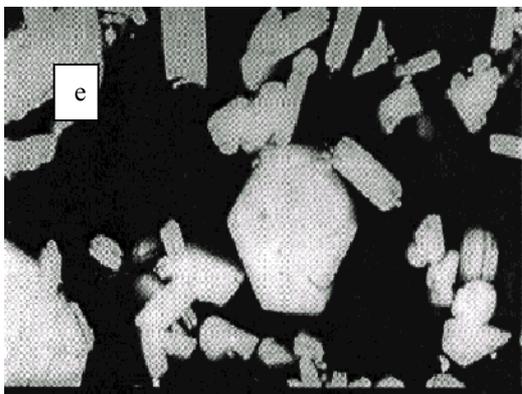
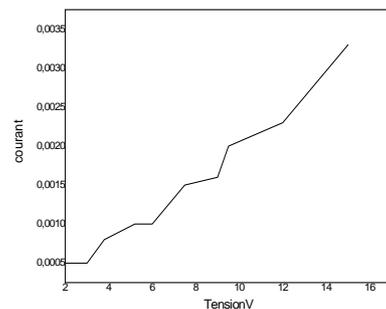
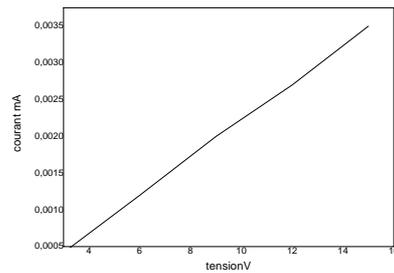


Fig 7: Scanning electron micrograph of $SnNb_5Se_9$, PANI/ $SnNb_5Se_9$.

3.4 .Electrical properties

The results of electrical conductivity from the four points of new compounds resulting demonstrate the behavior of electrical properties of these compounds, and with a different conductivity of PANI therein to a value $2S / cm$ [16].



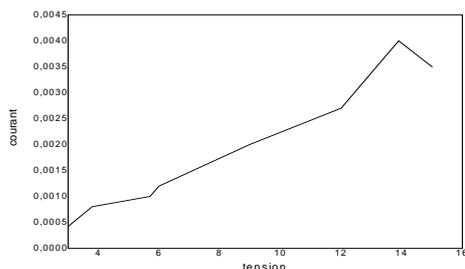


Fig 8 : represent mesure the conductivity electric of new compound

CONCLUSION

This work is based on two well known families of compounds, inorganic compounds and polymers, with which we have found the possibility of mix to build new hybrid organic inorganic phase. The addition of an inorganic compound in the matrix of polyaniline (polymer) is possible thanks to a particular synthesis method, and allows a new hybrid material. The study was conducted in states that X-ray diffraction spectra PANI/NbSe₂, PANI/Nb₃Sn and PANI / SnNb₅Se₉ are resulting from the addition of two families, morphology and the study confirms the structural study. Finally, we conclude the work by measuring the electrical conductivity of these products.

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