



# ICXRI

2006

International Conference  
on X-Ray and Related Techniques  
in Research and Industry

**PROGRAMME BOOK**

Palm Garden Hotel  
Putrajaya

Organiser



Co-Organiser





# ICXRI 2006 Conference Programme

## Day 1: 29 November 2006

Time	Venue	Activity
8.00 – 9.00	Foyer	Registration
9.00 – 9.10	Perdana B	<b>Chairman:</b> Assoc. Prof. Dr. Fauziah Haji Abdul Aziz (UMS)  Welcome & Conference Introduction by Professor Dr. Mohd Ambar Yarmo, ICXRI2006 Chairman cum XApp-MNS President
9.10 – 10.00	Perdana B	<b>Chairman:</b> Assoc. Prof. Dr. Fauziah Haji Abdul Aziz (UMS)  Keynote Lecture – Dr. Nahrul Khair Alang Md. Rashid, Deputy Director General (R&D) MINT and President Malaysian Nuclear Society. "X-ray Applications in Research & Industry: Malaysian Scenario"
10.00 – 10.30		<b>Tea Break &amp; Photo Session</b>
10.30 – 11.15	Perdana B	<b>Chairman:</b> Professor Dr. Mohd Ambar Yarmo (UKM)  Invited Lecture 1 Prof. Hans-Joachim Freund (Germany) <b>"Ultra-Thin Film Oxides: Materials With Tailored Properties"</b>
11.15 – 12.00		Invited Lecture 2 Martin Schreyer, Dr. rer. nat (Ph.D), M.Sc. (Nanyang Technological University, Singapore) <b>Unusual Particle Size Distribution in High Temperature Synthesized CeO<sub>2</sub></b>
12.00 – 12.45		Vendor's Presentation 1 Dr. Aleksandar Stefanovic (Panalytical Singapore)  <b>X-ray diffraction: from research to process control</b>
12.45 – 2.15		Lunch & Zohor

## Parallel Sessions Scientific papers presentation (2.15 pm – 5.15 pm)

Time	Parelel 1A (Room: Perdana A)  Chairman: Dr Hazizan Md Akil (USM)	Parelel 1B (Room: Perdana B)  Chairman : Associate Prof. Dr. Sahidan Radiman (UKM)	Parelel 1C (Room: Putra 3)  Chairman: Dr. Badrol Ahmad (TNBR)
2.15 - 2.35	<b>The Molecular Structure of [Ho(Pic)<sub>3</sub>(OH)<sub>2</sub>]<sub>3</sub>.2(18C6).4H<sub>2</sub>O Complex</b>  Eny Kusriani, Muhammad. I. Saleh, Bahrudin Saad, Rohana Adnan, H.-K. Fun, Bohari M. Yamin (USM)	<b>Stability of Dolomite Under Extreme Hetaing Condition: Comparison Between Perlis and Ipoh Dolomite</b>  Farizul, H.K., Mohd Nazry, S., Suhardy, D., Saiful Azhar, S. (KUKUM)	<b>An Investigation of Graphite like Carbon in Bangladesh Coal by XRD and Associated Raman Spectroscopic Techniques</b>  Jiban Podder and Tafazzal Hossain (BUET)
2.35 – 2.55	<b>A New Observation on the HEMT Structures Respect to the Change of Electrical Properties Using HRXRD Technique</b>  Idris Sabtu, Hariyadi Soetedjo, Mohamed Razman Yahya and Awang Mat A.F., (TMRND)	<b>Structural and Morphological Study of Nanocrystalline Nickel-Copper Particles</b>  Gafar Hayder Moh. Saeed, (UKM)	<b>Microstructure Characterization of Mechanically Activated Nanocrystallite Quartz using XRD Line Broadening</b>  Sammayamuththirian (USM)
2.55 – 3.15	<b>Lattice Constants Analysis of Ba<sub>0.3</sub>Sr<sub>0.7</sub>TiO<sub>3</sub> Ceramic for x = 0.3; 0.5 and 0.7 by Delphi Program</b>  Z. Jamal, Irzaman, M.S.Idris, M. Barmawi (KUKUM)	<b>Effect of Oxygen Partial Pressure on the Vacuum-Treated and Oxidised Ti<sub>3</sub>SiC<sub>2</sub> Using Grazing-Incidence Synchrotron Radiation Diffraction</b>  Zeya Oo, I.M. Low, B.H.O'Connor, K.E.	<b>Study of Microfibrils in Acacia Mangium Wood using Small Angle X-Ray Scattering (SAXS)</b>  Tamer A. Tabet, Sahidan Radiman & Fauziah Bt Haji Abdul Aziz, (UMS)



		Prince and A.J. Atanacio (CURTIN UNIV SARAWAK)	
3.15 – 3.35	Investigation of $V_2O_5$ Size and Shape Effects Using XPS  N. Asim, S. Radiman and M.A. Yarmo, (UKM)	Structure of Rare-earth Phosphate Glasses by X-ray and Neutron diffraction  H.B. Senin, W.B. Wan Nik and W. Kancono (KUSTEM)	Study of Growth in $Bi_2Sr_2CaCu_2O_y$ single crystal  Razak Mphd Ali Lee & H. Katsuyoshi (UMS)
3.35 – 3.55	Water tolerant solid acid of supported zirconium sulfate on HZSM-5 for esterification of oleic acid  Joon Ching Juan, Jingchang Zhang, Yajie Jiang, Weiliang Cao and Mohd Ambar Yarmo (University of Chemical Technology, China)	Mineralogical Study of Clay Samples From North-West Peninsular Malaysia Using X-Ray Diffraction Method  Ahmat Saat, Zaini Hamzah (UITM)	Crystallites Dimension of Carbon Pellets From $H_2SO_4$ Treated Self-Adhesive Carbon Grains Prepared From Oil Palm Empty Fruit Bunch  Mohd. Hafizuddin Haji Jumali, Mohamad Deraman, Astimar Abdul Aziz, Ramli Omar, Rozan Mohamad Yunus, Mazliza Mohtar and Abu Baker Elshiekh Abdelrahman (UKM)
3.55 – 4.15	Performance Analysis of Carbon Doped Tin Oxide Anode Material For Lithium – Ion Batteries  Hasanally, S.M. and Bustam M. Azmi, (SIRIM)	Modulation Transfer Function (MTF) Study on Amorphous Silicon Flat Panel System For Industrial Digital Radiography  Khairul Anuar b. Mohd Salleh, Ab. Razak b. Hamzah and Mohd Ashar Hj. Khalid (MINT)	Study of Anodised PM Aluminum Matrix Composite Reinforced with 15 wt% Saffil™ Alumina Short Fibre  Mohd Nazree Derman, Zainal Arifin Ahmad, Luay Bakir Hussain and Nurulakmal Mohd Sharif (USM)
4.15 – 4.35	Synthesis of CuS Nanoparticle Using Microemulsion Method  Muhammad Azmi Abdul Hamid & Agnes Ng Wei (UKM)	Production of Titanium Carbide by Thermal Explosion and TiC/Ti Functionally Graded Materials (FGMs)  Rahbari R. , R. Yahya, Dr. M. Hamdi, Farhudi O. (UM)	Microstrain Particle Size and Lattice Constant of $CaCO_3$ Ceramic by Rietveld Analysis  Irzaman, Z. Jamal, M.S. Idris, D. Kurnia, M. Barmawi (KUKUM)
4.35 – 4.55	Characterization of <i>Polymesoda expansa</i> (lokan) Mollusk Shell  Rosnah Nawang and Mohd Zobir Hussein, (UPM)	Structural and Electrical Characteristic of $Ba_{0.7}Sr_{0.3}TiO_3$ with a Secondary Phase $BaTi_2O_5$  Srimala Sreekantan, Ahmad Fauzi Mohd Noor, Zainal Arifin Ahmad, Radzali Othman, Anthony West, Derek Sinclair (USM)	Low Angle X-Ray Diffraction and Diffuse X-Ray Scattering Study of Interface Structure in Co/Cu Multilayers  K.Y. Kok and I.K. Ng (MINT)
4.55 – 5.15	Spectroscopic studies of cow femurs and <i>Porites Ramosa</i> coral from Sabah  Fadhilia Zafarina Zakaria and Fauziah Bt Hj Abdul Aziz (UMS)	Influence of Milling on Hydroxyapatite Particles  Sofea Beagem Mohd Noal, Roslinda Shamsudin, Tan Lee Phin and Wan Kartini Wan Abdul Kadir (UKM)	The Study of Nanosized Nb Doped Tetragonal Y-ZrO <sub>2</sub> Powders  Niki Prastomo, Zainovia Lockman, Ahmad Nuruddin, Atsunori Matsuda, Ahmad Fauzi Mohd Noor (USM)
5.15	End of 1 <sup>st</sup> Day and Tea		
8.15 – 11.00	Official Opening and Conference Dinner at Perdana B		



12.40 – 12.40	<b>In-situ XRD Study on Phase Transformation of Alumina</b>  Meor Yusuff M.S. and Masliana Muslimin (MINT)	<b>Nanostructure Studies of Charge Transfer Complex (CTC) in Powder of Terthiophene Bisillated and TCNQ by X-Ray Diffraction</b>  Kanco, H.B. Senin, Ku Halim Ku Bulat, (The Univ of Bengkulu, Indonesia)	<b>Field Emmission of Nitrogen Doped Diamond Like-Carbon (DLC) Thin Film</b>  Kahirul Anuar Mohamad & Naoki Okuyama (UMS)
12.40 – 2.15	<b>Lunch &amp; Zohor</b>		
<b>Time</b>	<b>Venue</b>	<b>Activity</b>	
2.00 – 3.30	<b>Perdana C</b>	<b>Poster session</b>	
3.30 – 4.00	<b>Perdana B</b>	<b>Chairman: Dr. Abdul Aziz Mohamed (MINT)</b>	
4.00 – 4.30		Invited Lecture 6 Prof. Bokhari Yamin (UKM) <b>Chemical analysis and X-ray Crystallography</b>	
4.30 – 5.00		Invited Lecture 7 Prof. Fun Hoong Kun (USM) <b>Hydrogen bonding phase transition in some organic crystals</b>	
5.00	<b>Perdana B</b>	Invited lecture 8 Assoc. Prof. Dr. Sharifah Bee (UM) <b>X-ray powder diffraction – Traditional applications and current advances</b>	
		<b>Conference Closing Ceremony and High Tea</b>	



## NANOSTRUCTURE STUDIES OF CHARGE TRANSFER COMPLEXES (CTC) IN MATERIALS OF TERTHIOPHENE BISILYLATED AND TCNQ BY X-RAY POWDER DIFFRACTION

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### ABSTRACT

Charge transfer complexes (CTC) can be readily introduced into nanomaterials by cohydrolysis-copolymerisation of bis-silylated terthiophenes with TMOS and TEOS in the presence of tetracyanoquinodimethane (TCNQ). CTC formation was shown in the vibration spectra that strong vibration  $C\equiv N$  in 2184, 2120 and 1595  $\text{cm}^{-1}$  as peaks characteristics of CTC. The study using EDAX-SEM shows that there are presence of spheres rich in silicon in the case of the gels prepared in the presence of TCNQ by the diameter 5  $\mu\text{m}$ . These spheres are eliminated after washing by acetone and remain the sphere with the diameter less than 5  $\mu\text{m}$ . These phenomena have shown that the TCNQ contributed to construct of the nanostructure in the formation of CTC. Finally, the organization structure of the CTC in that powder material has been studied by the XRD and the SAXS. In this case there is an organization as a lamellar structure was observed for xerogel [BTS3T-TCNQ-6TMOS], furthermore after this powders being washed by acetone the structure was disappeared as shown by the diffractogram of SAXS as shown by diffractogram of SAXS in fig.5. The peaks at 5.9, 5.6 and 4.0  $\text{\AA}$  were disappeared, except the peak at 4.6  $\text{\AA}$  as the matrix for Si-O-Si, which is still present after the gel being washed with acetone. This phenomena has been concluded that TCNQ have contribute to CTC formation in the matrices of silsesquioxane network of silylated terthiophene  $[\text{O}_{1.5}\text{Si}-(\text{C}_4\text{H}_2\text{S})_3-\text{SiO}_{1.5}]_n$ .

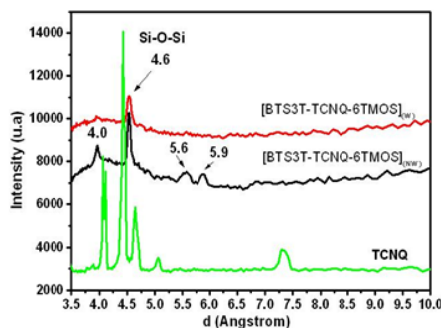


Fig. 5. Diffractogram of xerogel [BTS3T-TCNQ-6TMOS]<sub>NW</sub> and [BTS3T-TCNQ-6TMOS]<sub>W</sub>.



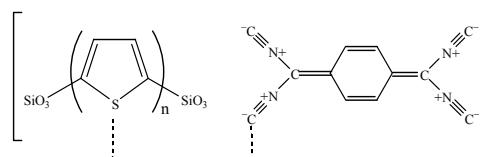
## I. INTRODUCTION

Charge transfer complexes (CTC) is the phenomenon of electron transfer from a compound or species that have some probability that the passage of electrons transferred to other compounds. This phenomenon makes a common way to perform the characterization of compounds that have electrical properties. In the organic-inorganic polymer material can be used as a method for studying the physical and chemical properties of a material [10, 11].

A new material to be learned from organosilicon physical properties and chemical properties, especially the structure of compounds formed. In the compound hybrid organosilicon generally present the basic characteristics of the known, which is amorph. However semi-crystalline forms are often found, when processed by a technique and a specific method. The method is widely used is the dry method and wet method. Both of these methods differ only in preparation, dry method performed without the use of fluids, but direct the formation of polymer ceramics through condensation and then proceed with sintering and heating. While the wet method using a solvent for the reaction between compounds that bond with each other [1].

Preparation of a new form of ceramic material of silicon derived from the hydrolysis and condensation reaction between the organosilane compound TMOS (tetramethoxysilane) or TEOS (*tetraethoxysilane*) is an amorphous material. However, the presence of precursors containing thiophene ter-sililasi methoxy ( $\text{CH}_3\text{-O-}$ ), amorphous nature can be reduced. This has been studied and observed from its nature, where the value of birefringence for thiophene ter-sililasi very high value, which is  $3 \times 10^{-9}$  [7, 10, 11].

In the present study will examine the effect of nanostructures on the CTC of an electron-rich molecules sililated terthiophene with *methoxysilane*  $[(\text{CH}_3\text{-O})_3\text{-Si}]$  using compound *tetracyanoquinodimethane* (TCNQ) containing the chromophore and has the ability to interact on a bond double ( $\text{C}=\text{C}$ ) or triplicate ( $\text{C}\equiv\text{N}$ ) which contains  $\pi$ -delocal orbital, which has the possibility for interaction and movement of electrons from the orbital to another orbital more dynamic [7, 10, 11].



**Figure 1.** The Charge Transfer Complexes (CTC) between terthiophene with TCNQ

The study about phenomenon of nano-structured compound CTC organosilicon requires some theoretical device of X-Ray and Scanning Electron Microscopy. Some equipment is in principle be used to study nanostructures were: X-Ray Diffraction, Small Angle X-Ray Scattering (SAXS) and Scanning Electron Microscopy (SEM) [7, 10, 11].

SAXS is a detection device structure using X-ray beam source stand-alone, data analysis, SAXS measurements on many samples to use to materials that contain nanoparticles. The sample is composed exclusively of the dispersion of particles in liquid, powder nano-size materials (nanopowders), and nanocomposites. It is also used for the evaluation of the data scattering of particles of a solid structure of mesoporous materials [8, 9].



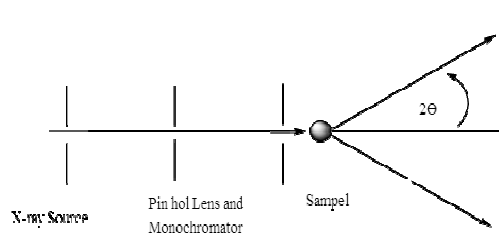
SAXS is a technique widely used to characterize the structure of solids and liquids in the range of nanometer (nm). Analysis using a probe that a majority of homogeneous but there are very few impurities (inhomogeneities), so it can operate from the electron density on length scales of 1-100 nm. Data generated diffractogram of structural information to complement the data XRD (WAXS-angle X-ray scattering width). So how this applies to the characterization of crystalline materials (polycrystalline, semicrystalline) and amorphous [9, 10].

Measurements are usually performed in a transmission geometry using a cylindrical capillary tube and X-ray sources. Angle scattering is usually between 0.1 and 5 degrees. Some specifications of SAXS capabilities are: (1) Analysis and quantification - determine the crystal structure. (2) Transmission - Convert transmission system geometry, so that the peak position can be determined more accurately and the intensity at low angles. (3) and Thin Film Reflectivity analysis - measuring the thickness, quality, and the phase of the layer structure of thin films. (4) Angle X-ray scattering is very small (Small Angle X-ray Scattering), an ideal way to characterize nano-sized structures. Features (artifacts composition of a particle) can be accessed from the position of the sharp little corners. Samples can be prepared and measured in a cylindrical capillary, which is made of a thin layer of polymer [8, 9].

The usefulness of X-ray diffraction powder for the identification of atoms in a solid crystalline phase of 3-dimensional, is widely used in many fields, among others: (1) The identification of minerals in geological samples, help understand the mechanism of formation of material which can provide information about the existence of ore. (2) Detection of polymorphs (=chemical structure is

similar, but different phases) of a given material-very important for the pharmaceutical industry. (3) Determination of the quality of the impurities in the pure phase to 0.1% by weight. (4) Corrosion in boilers by providing information about the condition and reactions that cause corrosion and giving instructions on how to minimize the corrosion process. (5) Forensic: phase identification can be a factor in determining the origin of traces found at the scene of the crime. (6) Analysis of the thin film, the particle size of nanomaterials, investigate the effect of temperature on material properties, automated analysis on some useful until the latest developments in optics, and software [8, 9].

Analyses of SAXS diffractogram is also able to calculate the values of the reference intensity ratio (RIR) and the scale factor of a phase, based on the intensity / peak area per phase. It is particularly suitable for the amorphous phase with irregular peak shape. X-ray diffraction scheme in Figure 2. The following provides an overview analogous to the formulation of  $d$  or  $q$  in SAXS diffractogram.



**Figure 2.** Scheme of X-ray Diffraction

SAXS analysis performed using X-ray source of synchrotron radiation to have a wavelength  $\lambda = 0.1608$  nm. Based on the Bragg formula,  $d = \lambda \sin 2\Theta$ , then with small  $\Theta$ , then the vector scattering is  $\Theta/2$  and the distance between the lattice



denoted by  $q$ , where the distance was assumed with a density between particle / atom. In the SAXS beam arrangement used to use one set of plates of silicon monochromator with a predetermined gap in geometry. Density or the distance between the particles ( $q$ ) has a minimum limit that is assumed equal to the density ( $\rho$ ) which can be measured with a mercury picnometer, with units of ( $\text{nm}^{-1}$ ). One-dimensional position of the detector is set by channel length of  $6.49 \times 10^{-2}$  mm/canal, used to record the SAXS intensity as a function of scattering vector  $q$ , so that the Bragg formula is modified according to Guinier formula where the distance is calculated as  $q = (4\pi/\lambda)\sin(\Theta/2)$ , where  $\lambda$  is the wavelength of radiation and  $\Theta$  is the scattering angle for small angle is  $(\Theta/2)$ . Sample with a detector distance of 755 mm set, which allows us to investigate more thoroughly the distance  $q$ ,  $q_0 =$  range from  $0.19 \text{ nm}^{-1}$  to  $q_m = 4.4 \text{ nm}^{-1}$ , with a resolution of  $\Delta q = 3.36 \times 10^{-3} \text{ nm}^{-1}$  [5].

## II. METHODOLOGY

### A. Ingredients:

Precursor: 2,5-bis(trimethoxysilyl)-terthiophene (BTS3T) ; tetramethoxysilane (TMOS) and tetraethoxysilane (TEOS), terthiophene ( $\text{C}_4\text{H}_2\text{S}$ ), tetracyanoquinodimethane (TCNQ), solvent DMF (dimethylformamide), hexane, catalyst ammonium fluoride ( $\text{NH}_4\text{F}$ ; 0.1M), all of the material obtained from E. Merck.

Polar solvent for washing:  $\text{H}_2\text{O}$  (aquades-tilata), alcohol, and acetone.

### B. Apparatus:

Reactor for the preparation; hotplates, paraffin bath, inert Nitrogen gas lines, test tubes, manometers, vacuum compressors, syringe 10 ml, Heater (hair dryer), mortar mortar and pestle, stainless stirrer.

Characterization tools: X-Ray specimen holder, holder X-Ray Diffraction, X-Ray Diffraction, SAXS capillary cylinder,

Small Angle X-Ray Scattering (SAX), Scanning Electron Microscopy (SEM), mortar, spatula.

### C. Procedure:

#### Preparation

Samples prepared in 3 test tubes with paraffin heaters in the hotplates a porous material made of the hydrolysis reaction and polycondensation of compounds with varying ratios: the precursor 2,5-bis(trimethoxysilyl)terthiophene (BTS3T) with TCNQ (0.1 mgr in 5 ml of DMF) added tetramethoxysilane (TMOS) (98%) (varies from 1, 3 and 6 mol), then 10 ml distilled water, and 1 ml of 0.1 N  $\text{NH}_4\text{F}$  as the catalyst. Then the pH of the mixture is set approximately 1.5 to 2.0. Hydrolysis carried out for 10 minutes under a constant temperature of  $60^\circ\text{C}$  and 20 kHz ultrasonic radiation with a constant power (60W). Sol solution formed in the mixture allowed to stand 10 minutes for cooked and perfectly homogeneous, with the granting of  $\text{NH}_4\text{OH}$ , acidity, or pH of the sol is set at 4.5 to 5.0 [3, 4]

The process of gel formation will occur within a few minutes, then take this gel is formed and heat until the solvent evaporates, and formed a porous silisium xerogel materials. Xerogel stored in closed containers for gelation and aging (aging) is maintained for 10 days at  $60^\circ\text{C}$ . After drying, forming porous ceramics known as xerogel. These samples were crushed with a mortar and prepared for analysis of density (density), the analysis of the structure and morphology by SAXS and SEM.

### D. Analysis:

#### X-Ray Diffraction and SAXS

Samples at each end of the period of aging (aging) with a constant temperature heating of samples taken out of the tube and slowly cooled to room temperature for analysis of density and SAXS. Each sample was crushed with a mortar are



labeled according to the constancy of temperature, ceramic materials are put into a cylindrical capillary SAXS. Install a capillary filled cylinder at the center of the holder SAXS.

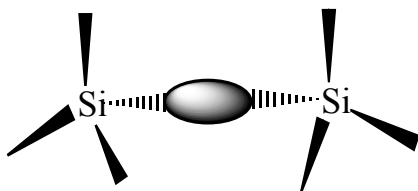
Equipment used SAXS: SAXS is available at laboratory material the University of Montpellier .

### SEM:

SEM tool that is used as a comparison and strengthen analisis morphology available in the laboratory of Biology at University of Montpellier II, France.

## III. RESULT AND DISCUSSION

Material generated from these experiments a xerogel which had charge transfert complex (CTC) with the composition: [BTS3T-TCNQ-6TMOS], where the network is formed as a silsesquioxane  $[O_{1.5}Si-(C_4H_2S)_3-SiO_{1.5}]_n$ . Structurally can be described as followsscheme:



**Figure 3.** Scheme silsesquioxane matrix structure of the tetrahedral network  $[O_{1.5}Si-(C_4H_2S)_3-SiO_{1.5}]_n$

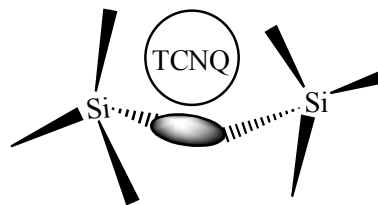
Data is based on the behavior of the scattering intensity of SAXS Porod region can be concluded that the xerogel at 60 °C proved to have a fractal surface structure with fractal dimension 2.5 nm, the length scale between  $1/q \sim 1.5$  and  $1/q \sim 0.5$  nm. This means that the process of formation of solids at this stage may occur emptying of the pores. Thus, the figure. 2 shows a relatively small decrease in density. The average size of the pores are determined by the radius range is reduced

gradually decreased from samples containing element particles of *tetra-cyanoquiodimethane* (TCNQ).

**Table 1.** Xerogel composition and density before and after washing

No	Composition (mol)			Density (S/V)	
	BTS3T	TCNQ	TMOS	Before washing	After washing
1	1	-	2	0,6	0,5
2	1	1	2	1,0	0,8
3	1	-	4	1,2	1,1
4	1	1	4	1,3	1,2
5	1	-	6	1,8	1,2
6	1	1	6	2,0	1,2
7	1	-	8	1,8	1,4
8	1	1	8	2,4	1,3

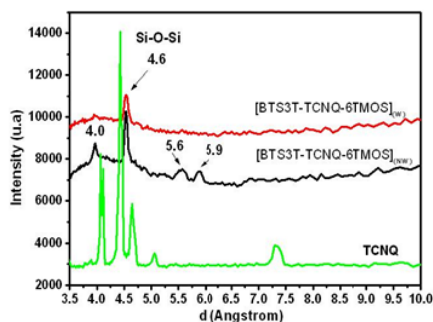
Table 1.shows the structural parameters associated porosity, surface area per unit volume of the sample, the S/V, determined S/m, through the relation  $S/m = (1/\rho)S/V$ . This means that the material in the presence of TCNQ are used for the CTC, the pores are formed between the matrix of Si-O-Si-O and polymer plates thiophene ( $C_4H_2S$ ) is made such that the density is increasing, and the washing or removal of TCNQ occurs shrinkage due to particle TCNQ has been eliminated in such a way, where the pores of the matrix as the artifacts left behind and will be constantly increasing in accordance with the original structure.



**Figure 4.** BTS3T nanocomposites scheme in the presence of TCNQ (filling the space between the tetrahedral geometric).

From the table it is shown that sintering will also affect the surface properties of nanopores which disappeared into mesoporous after washing TCNQ.





**Figure. 5.** Diffractogram of xerogel [BTS3T-TCNQ-6TMOS]<sub>NW</sub> and [BTS3T-TCNQ-6TMOS]<sub>W</sub>.

From the intensity data from SAXS diffractogram is shown that the maximum intensity of TCNQ is present in 4.0 and 4.6 angstroms, appeared on the xerogel-CTC in 4.0; 4.6; 5.6 and 5.9 angstrom and found that the peak TCNQ decrease after xerogel are washing, but still left the pore diameter (q) 4.6 angstroms. The SAXS intensity can be concluded that the structure in the presence of CTC is significantly strong enough. It also shows that xerogel structure formed of the solid structure with a lamellar order.

#### IV. CONCLUSION

Charge transfer complexes (CTC) can be readily introduced into nanomaterials by cohydrolysis-copolymerisation of bisilylated terthiophenes with TMOS and TEOS in the presence of tetracyanoquinodimethane (TCNQ). CTC formation was shown in the vibration spectra that strong vibration  $C\equiv N$  in 2184, 2120 and 1595  $cm^{-1}$  as peaks characteristics of CTC. The study using EDAX-SEM shows that there are presence of spheres rich in silicon in the case of the gels prepared in the presence of TCNQ by the diameter 5  $\mu m$ . These spheres are eliminated after washing by acetone and remain the sphere with the diameter less than 5  $\mu m$ . These phenomena have shown that the TCNQ contributed to construct of the nanostructure in the formation of CTC.

Finally, the organization structure of the CTC in that powder material has been studied by the XRD and the SAXS. In this case there is an organization as a lamellar structure was observed for xerogel [BTS3T-TCNQ-6TMOS], furthermore after this powders being washed by acetone the structure was disappeared completely.

The peaks at 5.9, 5.6 and 4.0 Å were disappeared, except the peak at 4.6 Å as the matrix for Si-O-Si, which is still present after the gel being washed with acetone. This phenomena has been concluded that TCNQ have contribute to CTC formation in the matrices of silsesquioxane network of silylated terthiophene  $[O_{1.5}Si-(C_4H_2S)_3-SiO_{1.5}]_n$ .

#### Acknowledgements

This research is largely supported by the laboratory of Prof. Corriu and Prof. Andre Vieux and Laboratorium of XRD/SAXS nanomaterial, with technicians ; Mr. Dieudone at the University of Montpellier II, France.

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