

Research Article

Synthesis and Characterization of a New Hybrid Polyoxometalate Compound: Bis(3-aminopropyl)ammonium Hexatungstotellurate(VI) Octahydrate

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Abstract

Synthesis strategy of hybrids Polyoxometalates have drawn a lot of attention owing to interesting. Herein, we describe, the one pot process used to synthesize a new hybrid polyoxotungstate, $(C_6H_{20}N_3)_2[TeW_6O_{24}].8H_2O$, at a temperature of 200 °C. This preparation was carried out by mixing equimolar amounts of H_2WO_4 , $C_6H_{17}N_3$, and H_6TeO_6 . The compound was characterized by infrared and UV-visible spectroscopy techniques, as well as by X-ray diffraction. The compound crystallizes in the monoclinic system with the space group $P2_1/c$. The unit cell are: $a = 8.74874(14) \text{ \AA}$, $b = 12.5894(2) \text{ \AA}$, $c = 18.5176(3) \text{ \AA}$, $\alpha = 90^\circ$, $\beta = 92.6078(14)^\circ$, $\gamma = 90^\circ$. The compound consists of a non-protonated Anderson-type heteropolyanion $[TeW_6O_{24}]^{6-}$ stabilized by two organoammonium cations $[C_6H_{20}N_3]^{3+}$ and eight water molecules. The polyoxoanion $[TeW_6O_{24}]^{6-}$ adopts an Anderson-Evans type structure of class A. Thus, the polyanion is built of six $\{WO_6\}$ units surrounding a $\{TeO_6\}$ octahedron. The six $\{WO_6\}$ are linked together by edges sharing and connected around the heteroatom, via oxygen atoms, forming a planar structure with approximate D_{3d} symmetry. The cohesion of the three-dimensional structure is ensured by hydrogen bonds between the polyanions, the organoammonium groups, and the water molecules, thereby providing significant stability to the compound. UV-Visible absorption spectroscopy shows a strong absorption band at 296nm attributed to Ligand-Metal Charge Transfer (LMCT) transition of the $O \rightarrow W$ and the main IR absorption bands of the polyanion appear at: 942 cm^{-1} ; 875 cm^{-1} ; 766 cm^{-1} and 599 cm^{-1} .

Keywords

Anderson-type Polyoxometalate, Hybrid, Hydrogen Bonds, Organic Counterions

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1. Introduction

The Anderson-Evans type polyoxometalates (XM_6O_{24}) represent a captivating category of polyanions with a complex and diverse structure [1]. These clusters consist of an XO_6 unit surrounded by six MO_6 groups ($\text{M}=\text{W}$ or Mo) adopting an octahedral geometry. Their remarkable stability and unique physicochemical properties make them distinctive entities. Consequently, these compounds have sparked growing interest within the inorganic chemistry community due to their potential in catalysis [2-6], biology [7, 8], and optics [9, 10]. The cage-like configuration of their structure offers a wide range of functionalization and modulation possibilities, thus paving the way for multiple applications in various fields of scientific research.

The Anderson-Evans type polyoxomolybdates are divided into two distinct groups: the non-protonated type A, with the general formula $[\text{X}^{n+}\text{M}_6\text{O}_{24}]^{(12-n)-}$, and the protonated type B, with the general formula $[\text{X}^{n+}(\text{OH})_6\text{M}_6\text{O}_{18}]^{(6-n)-}$. Practically all elements from the p and d blocks can act as heteroatoms, and there are more polyoxomolybdates of Anderson-Evans type than polyoxotungstates of the same type. The latter play a crucial role in polyoxometalate chemistry and are usually synthesized from an isopolyoxotungstate or tungsten oxide. For example, Oreshkina et al. [11] described a purely inorganic compound, $[\text{Na}_2(\text{H}_2\text{O})_{10}][\text{Na}(\text{H}_2\text{O})_3]_2[\text{MnW}_6\text{O}_{18}(\text{OH})_6] \cdot 6\text{H}_2\text{O}$, containing a polyanion $[\text{MnW}_6\text{O}_{18}(\text{OH})_6]^{4+}$, while Schmidt and Schrobilgen [12] developed $\text{Na}_6[\text{TeW}_6\text{O}_{24}] \cdot 22\text{H}_2\text{O}$. Furthermore, literature reports other hybrid [13-15] and purely inorganic [9, 16-18] Anderson-Evans type polyoxotungstates.

In this line, we present the compound $(\text{C}_6\text{H}_{20}\text{N}_3)_2[\text{TeW}_6\text{O}_{24}] \cdot 8\text{H}_2\text{O}$, an organic-inorganic hybrid polyoxotungstate of Anderson type.

2. Materials and Methods

2.1. General

The UV-visible absorption measurements of the compound were recorded using a Thermo Scientific GENESYS 10S UV-Vis spectrophotometer at room temperature in acidic aqueous solution (H_2SO_4).

IR spectroscopy measurements were carried out for the compounds. IR measurements were performed using ATR (Attenuated Total Reflectance) method from 4000 to 400 cm^{-1} [19].

A single-crystal X-ray diffraction data for the compound was measured on a Rigaku Oxford.

Diffraction diffractometer at the MoK α radiation. Data collection reduction and multiscan ABSPACK correction were performed with CrysAlisPro (RigakuOxford Diffraction). The crystal structures, including the anisotropic displacement parameters, were refined for both compounds with SHELXL-2018/3 [20-22]. Information Files were compiled

with Olex2.12. Crystallographic data are summarized in Table 1.

2.2. Synthesis

Tungstic acid H_2WO_4 (98%), bis(3-aminopropyl)amine $\text{C}_6\text{H}_{17}\text{N}_3$ (98%), and telluric acid H_6TeO_6 (99%) were purchased from Sigma-Aldrich and used without further purification with distilled water.

Functionalization by self-assembly method with one pot synthesis process was carried to obtain the compound.

The $(\text{C}_6\text{H}_{20}\text{N}_3)_2[\text{TeW}_6\text{O}_{24}] \cdot 8\text{H}_2\text{O}$ was obtained by mixing, in 40mL of distilled water, 0.999g of tungstic acid (H_2WO_4), 0.541g of bis(3-aminopropyl)amine ($\text{C}_6\text{H}_{17}\text{N}_3$) and 0.928g of telluric acid (H_6TeO_6). The resulting solution was heated under reflux and stirred for two hours. After a few days of slow evaporation at room temperature, colourless crystals suitable for X-ray diffraction were obtained.

3. Results

3.1. IR and UV-vis Spectroscopies

The infrared spectra of the compound highlight, in the range from 600 to 900 cm^{-1} , the stretching vibrations of the W-O-W and Te-O bonds of the polyoxoanion. The band corresponding to the stretching frequencies of the W=O bonds is located between 920 and 1000 cm^{-1} . As for the characteristic bands of the counter-anion, the deformation frequencies $\delta(\text{C-H})$ and $\delta(\text{N-H})$ are observed in the range of 1300 to 1600 cm^{-1} , while the frequency band $\nu(\text{O-H})$ is detected around 3000 cm^{-1} (Figure 1).

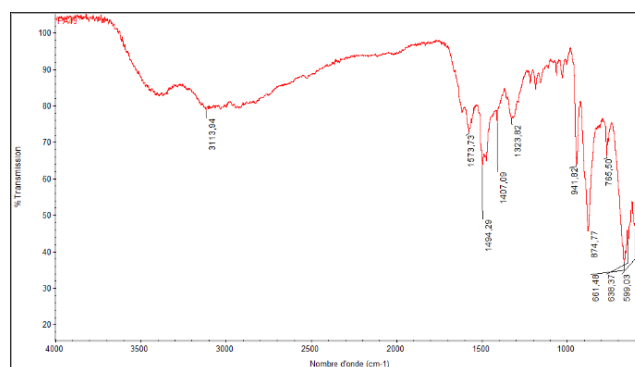


Figure 1. IR spectrum of the compound.

The UV-Visible absorption of the compound was analyzed in the 250-800 nm range in sulfuric acidic solution. The UV spectrum reveals precisely absorption band at 296nm for the compound (Figure 2). This absorption band is attributed to Ligand-Metal Charge Transfer (LMCT) transition of the

O→W [23-25].

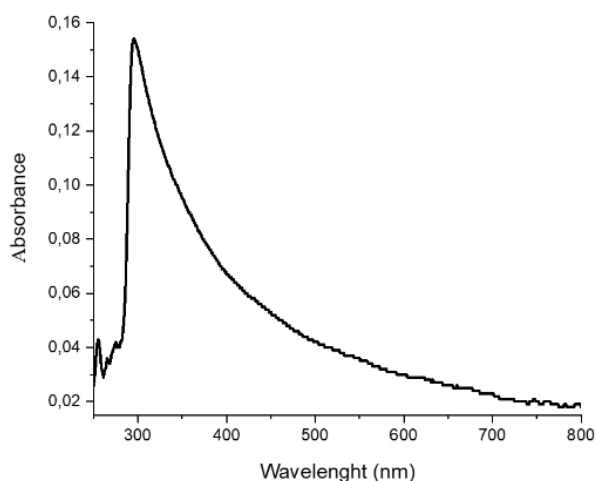


Figure 2. UV visible absorption spectrum of the compound.

3.2. Crystallographic Study

The crystallographic data was collected using X-ray diffraction analysis on a single crystal. This compound crystallizes in the monoclinic space group $P2_1/n$.

Table 1. Crystal data and details of data collection.

Column1	Column2
Empirical formula	$C_{12}H_{56}N_6O_{32}TeW_6$
Fw	2027.32
Temperature (K)	293
Space group	$P2_1/n$
	8.74874(14)
a, b, c (Å)	12.5894(2)
	18.5176(3)
α, β, γ (°)	90, 92.6078(14), 90
V (Å ³)	2037.45(6)
Z	2
μ (mm ⁻¹)	17.678
Crystal size (mm)	0.25 × 0.20 × 0.05
2 θ range (°)	3.914 to 50.046
Reflections collected	14105
Independent reflections, R_{int}	3588, 0.0522
Data, restraints, parameters	3588, 0, 274
Goodness-of-fit on F^2	1.088

Column1	Column2
R_1, wR_2 for $[I \geq 2\sigma(I)]$	0.0305, 0.0708
R_1, wR_2 for all data	0.0342, 0.0730
$\Delta\rho_{max}, \Delta\rho_{min}$ (e Å ⁻³)	1.61, -1.72

The asymmetric unit, as shown in Figure 3, comprises one heteropolyanion $[TeWO_{12}]^{3-}$, one organic counter-cation $[C_6H_{20}N_3]^{3+}$ and four interstitial water molecules.

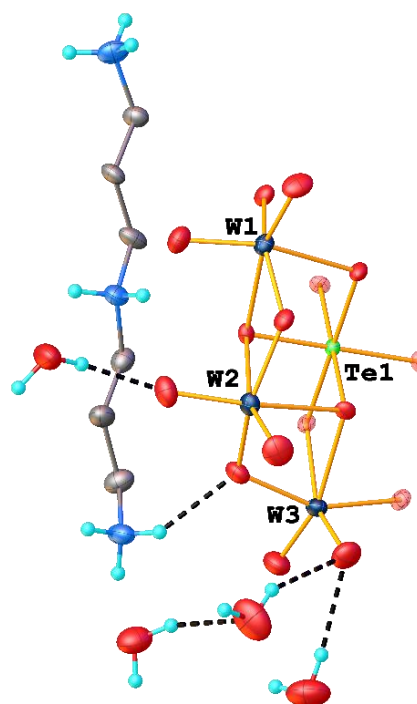


Figure 3. View of the asymmetric part in the crystal structure of $(C_6H_{20}N_3)[TeW_3O_{12}] \cdot 4H_2O$ showing the coordination environment of the metal atoms. The oxygen atoms with 50% translucent were generated by symmetry operation: $-x, -y, -z$. Non-relevant hydrogen atoms have been omitted for clarity.

The polyoxoanion $[TeW_6O_{24}]^{6-}$ adopts an Anderson-Evans type structure of class A, being a non-protonated Anderson-type polyoxotungstate. It consists of six $\{WO_6\}$ units surrounding a $\{TeO_6\}$ octahedron. These $\{WO_6\}$ monomers are linked together by shared edges and connected around the heteroatom via oxygen atoms (μ_3-O), forming a planar structure with approximate D_{3d} symmetry. Three types of oxygen atoms are present in this compound: terminal $W=O$ oxygens paired with each of the six tungsten atoms, double bridging oxygens (μ_2-O) ensuring the $W-O-W$ bonds, and triple bridging oxygens (μ_3-O) linking the heteroatom and two tungsten atoms. Various Anderson-type polyoxometalates have been described in the literature, such as purely inorganic compounds like $(NH_4)_3[AlMo_6O_{24}H_6] \cdot 7H_2O$ [26] and

$\text{Na}_4[\text{Ni}(\text{OH})_6\text{W}_6\text{O}_{18}] \cdot 16\text{H}_2\text{O}$ [27], as well as functionalized polyoxometalates like $\text{Na}_3\text{K}_3[\text{Cr}((\text{OCH}_2)_3\text{CC}_2\text{H}_5)\text{W}_6\text{O}_{21}] \cdot 17\text{H}_2\text{O}$ [14] and $\text{Na}_2[\text{TMA}]_2[\text{Ni}(\text{OH})_3\text{W}_6\text{O}_{18}(\text{OCH}_2)_3\text{CCH}_2\text{OH}] \cdot 9\text{H}_2\text{O}$ (TMA = tetramethylammonium) [28].

The tungsten (W) and tellurium (Te) atoms are hexacoordinated (Figure 4). The octahedron surrounding the tellurium atom is distorted due to its bonding with six $\mu_3\text{-O}$ oxygen atoms, resulting in bond lengths and angles different from those of a regular octahedron. Thus, the bond lengths and angles around Te vary from 1.967(4) to 2.267(5) Å and from 85.1(2) to 180(2) degrees, respectively. As for the tungsten atoms, they are bound to three types of oxygen: terminal oxygens have an average W-O_t bond length of 1.735 Å; $\mu_2\text{-O}$ oxygens have $\text{W}(\mu_2\text{-O})$ bonds slightly longer than W-O_t bonds, with an average of 1.928 Å; finally, the $\mu_3\text{-O}$ oxygens, bonded to two tungsten atoms, have an average $\text{W}(\mu_3\text{-O})$ bond length of 2.273 Å. These results are consistent with literature data [27, 29].

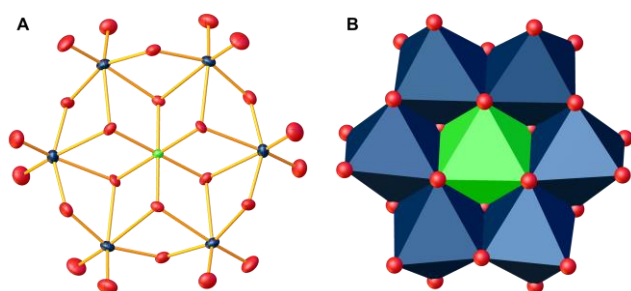


Figure 4. A view of the $[\text{TeW}_6\text{O}_{24}]^{6-}$ anion: A - thermal ellipsoidal representation at 50% probability level and B - polyhedral

Each component, the cation, anion, and interstitial water molecules, interact with each other through a system of hydrogen bonds that extends in all three directions, resulting in a complex three-dimensional network. A simplified view of this network is illustrated in Figure 5. These hydrogen interactions are summarized in Table 2.

Table 2. Hydrogen Bond parameters (Å, °).

$D\text{-H} \cdots A$	$d(D\text{-H})$	$d(H\text{-}A)$	$d(D\text{-}A)$	$D\text{-H-A}$
$\text{O1}_w\text{-H} \cdots \text{O12}$	0.88	2.12	2.942(9)	154.3
$\text{O1}_w\text{-H} \cdots \text{O3}^1$	0.89	2.13	2.904(8)	145.5
$\text{O2}_w\text{-H} \cdots \text{O1}_w$	0.85	2.01	2.811(9)	157.9
$\text{O2}_w\text{-H} \cdots \text{O2}^2$	0.84	2.10	2.931(8)	172.8
$\text{O3}_w\text{-H} \cdots \text{O7}$	0.90	1.88	2.729(8)	157.3
$\text{O3}_w\text{-H} \cdots \text{O5}^3$	0.86	2.03	2.888(7)	170.0
$\text{O4}_w\text{-H} \cdots \text{O11}^4$	0.85	2.09	2.810(9)	142.7

$D\text{-H} \cdots A$	$d(D\text{-H})$	$d(H\text{-}A)$	$d(D\text{-}A)$	$D\text{-H-A}$
$\text{O4}_w\text{-H} \cdots \text{O11}^5$	0.85	2.66	3.078(9)	111.9
$\text{O4}_w\text{-H} \cdots \text{O12}^5$	0.85	2.18	3.030(9)	174.6
$\text{N1-H} \cdots \text{O1}^6$	0.89	2.43	3.217(8)	147.4
$\text{N1-H} \cdots \text{O4}^6$	0.89	2.28	2.989(8)	136.5
$\text{N1-H} \cdots \text{O2}_w^2$	0.89	2.01	2.824(9)	152.1
$\text{N2-H} \cdots \text{O1}$	0.89	1.92	2.798(9)	170.4
$\text{N2-H} \cdots \text{O3}_w^7$	0.89	1.96	2.782(8)	152.6
$\text{N2-H} \cdots \text{O9}^8$	0.89	2.11	2.898(8)	147.5
$\text{N3-H} \cdots \text{O4}_w$	0.89	1.85	2.709(9)	162.8
$\text{N3-H} \cdots \text{O2}^4$	0.89	2.24	2.896(9)	129.8
$\text{N3-H} \cdots \text{O8}^2$	0.89	2.53	3.231(9)	136.4
$\text{N3-H} \cdots \text{O7}^2$	0.89	2.26	3.019(9)	142.5
$\text{N3-H} \cdots \text{O3}_w^2$	0.89	2.37	3.114(9)	141.2

¹ $\frac{1}{2} + x, \frac{1}{2} - y, -\frac{1}{2} + z$; ² $-x, 1 - y, -z$; ³ $-\frac{1}{2} + x, \frac{1}{2} - y, -\frac{1}{2} + z$; ⁴ $-\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; ⁵ $-\frac{1}{2} + x, \frac{3}{2} - y, \frac{1}{2} + z$; ⁶ $\frac{1}{2} - x, \frac{1}{2} + y, \frac{1}{2} - z$; ⁷ $-x, -y, -z$; ⁸ $\frac{1}{2} + x, \frac{1}{2} - y, \frac{1}{2} + z$.

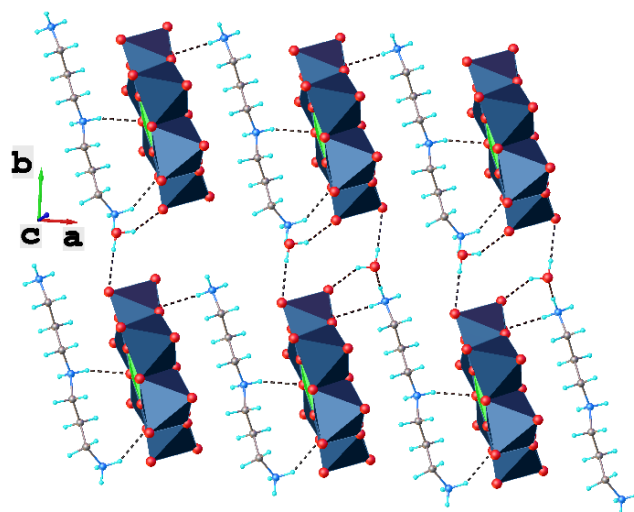


Figure 5. 3D supramolecular network viewed along c axis. The hydrogen bonds between the components are illustrated with dashed black lines.

4. Conclusions

A new inorganic-organic hybrid compound has been synthesized via one pot process and its structure has been characterized using X-ray diffraction (XRD), infrared spectroscopy, and UV-visible spectroscopy. The three-dimensional structure consists of polyanions, ammonium groups, and water molecules interconnected by hydrogen bonds. The bis(3-aminopropyl)ammonium groups are used to link the

polyanions to form layers, which are then linked to each other by water molecules through hydrogen bonds. These observations highlight the essential role of water molecules in stabilizing supramolecular assemblies.

The one pot synthesis method used in this work, compared to traditional thermal method, has various advantages. It requires no additional precursor; only basic reagents and it consumes less energy and requires a short reaction time. Additionally, crystals can be obtained in a single step.

In perspective, we believe that this diversity hydrogen bonding interactions between the polyanion, organic amines and water molecules and the built of edges sharing MO_6 octahedra in the polyanion type can lead to significantly different photochromic behaviors.

Abbreviations

Mo	Molybdenum
W	Tungsten
UV	Ultraviolet
TMA	Tetramethylammonium
LMCT	Ligand-Metal Charge Transfer
XRD	X-ray Diffraction
IR	Infrared

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Author Contributions

Papa Aly Gueye: Methodology, Writing – original draft
Lamine Yaffa: Supervision, Writing - review & editing
Dame Seye: Validation
S é rigne Fallou Pouye: Writing - review & editing
Alexandru-Constantin Stoica: Formal Analysis
Cheikh Abdoul Khadir Diop: Project administration
Mamadou Sidib é: Project administration

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Data Availability Statement

The data supporting the outcome of this research work has been reported in this manuscript.

Conflicts of Interest

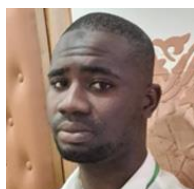
The authors declare no conflicts of interest.

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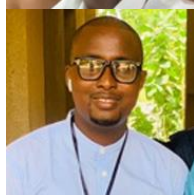
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Biography



Papa Aly Gueye, a doctoral student in chemistry at Cheikh Anta Diop University in Dakar UCAD, is a passionate researcher. He began his third cycle in 2020, after successfully completing his master's degree in molecular chemistry with a focus on inorganic chemistry in 2019. His master's thesis was on frustrated Lewis pairs, a complex topic that he mastered brilliantly. Currently, his research is focused on polyoxometalates and polynuclear complexes, fascinating areas of inorganic chemistry.



In 2022, **Lamine Yaffa** successfully obtained his Ph.D. in molecular chemistry at the Cheikh Anta Diop University. Renowned specialist in polyoxometalates and polynuclear complexes with numerous articles on these subjects. In 2016, he impressed during his master's thesis defense on antimalarial iron complex. Currently, he is focusing most of his research on clay sands, exploring new perspectives and advancing his understanding of chemistry.



Alexandru-Constantin Stoica is assistant research in the Department of Inorganic Polymers at “Petru Poni” Institute of Macromolecular Chemistry, Iasi, Romania, and defended his PhD thesis in 2023. He has experience in organic, inorganic, and material chemistry and routine characterization techniques. In terms of synthesis, he is particularly interested in the design of 2D coordination networks by using compounds containing a siloxane fragment that functions as both a ligand and a surfactant, with capacity of directing the planar development of the networks. Also, he has acquired skills in SC-XRD analysis.



Dame Seye, is a teacher-researcher, at the Iba Der Thiam University of Thiès, Department of Chemistry and Physical, UFR Science and Technology since 2021. He successfully obtained his PhD in inorganic chemistry at the Cheikh Anta Diop University of Dakar in 2019, as well as a Master's degree in molecular chemistry from the same institution in 2013. Dr. Seye is known for his unwavering commitment to research and teaching, as evidenced by his CAES diploma obtained at the faculty of sciences and education technologies at the Cheikh Anta Diop University of Dakar. His research, particularly his numerous articles on oxoanions and their complexes, makes him a key figure in the field. Currently, Dr. Seye is the head of the Licence and Master of Chemistry course at the Department of Chemistry and Physical, UFR Science and Technology, he continues to stand out for his expertise and passion for chemistry.



Serigne Fallou Pouye, a teacher-researcher within the prestigious Polytech school of Diamniadio, Departement of ingeniery Sciences and technics of Amadou Mahtar Mbow University. Dr Pouye has proven experience attested by a CAES diploma in educational sciences since 2013. He also taught physical sciences for ten years in secondary education. Dr. Pouye is an expert in the field of molecular chemistry. Holding a master's and a doctorate in inorganic chemistry obtained respectively from Cheikh Anta Diop University in Dakar in 2009 and 2012, Dr. Pouye has a wealth of experience in secondary education. With an impressive scientific output including more than a dozen articles, he was recruited as a teacher-researcher at UAM in 2020 and has since been serving as the head of the Department of Chemistry at the same university.



Cheikh Abdou Khadir Diop, professor at Cheikh Anta Diop University of Dakar, is a specialist in inorganic chemistry. Professor Diop is renowned for his significant advances in the field of research. He has actively contributed to numerous national projects. In 2012, he attained the enviable status of full universities professor, a well-deserved recognition of his expertise and commitment to higher education. Currently, he holds the position of assessor at the Faculty of Science and Technology at Cheikh Anta Diop University of Dakar, demonstrating his continued dedication to education and cutting-edge research.



Mamadou Sidibé is a prominent researcher and educator, a full professor at the prestigious Department of Chemistry at Cheikh Anta Diop University in Dakar. Professor Sidibé is recognized for his remarkable contributions to research. In 2018, he was honored as the dean of the Faculty of Technical Sciences, acknowledging his expertise and dedication to higher education. Professor Sidibé has played a key role in various collaborative projects both nationally and internationally, making him a key figure in the scientific community. Currently, he serves as the rector of Souleymane Niang University of Matam.

Research Field

Papa Aly Gueye: Research on inorganic materials chemistry

Lamine Yaffa: Research in inorganic chemistry, hybrid materials and Clay materials sciences

Dame Seye: Research on inorganic chemistry

Serigne Fallou Pouye: Research on inorganic chemistry

Cheikh Abdoul Khadir Diop: Research on inorganic chemistry

Mamadou Sidibé: Research on inorganic chemistry

Alexandru-Constantin Stoica: Research on inorganic chemistry