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RESEARCH ARTICLE

SYNTHESIS, SPECTROSCOPIC STUDIES (FTIR AND UV-VISIBLE), CRYSTAL STRUCTURE AND ELECTROCHEMICAL PROPERTIES OF A NEW INORGANIC-ORGANIC HYBRID MATERIAL BASED ON A B-OCTAMOLYBDATE CLUSTER $(C_{12}H_{28}N)_4[MO_8O_{28}].3H_2O$

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Abstract

A new organic-inorganic hybrid material based on a β-octamolybdate cluster$(C_{12}H_{28}N)_4[Mo_8O_{28}].3H_2O$,has been synthesized from ammoniu m heptamolybdate tetrahydrate $(NH_4)_6Mo_7O_{24}.4H_2O$, iodic acid HIO_3 and dihexylamine$[CH_3\ (CH_2)_5\ NH\ (CH_2)5CH3$ at 388K,then characteri zed by spectroscopic studies (FTIR and UV-Visible) and confirmed by single crystal X-ray diffraction (XRD). Thus, the redox properties of the compound were also been studied by cyclic voltammetry in H_2SO_4 (1M) at a scanning speed of 40 mV/s using glassy carbon as a reference electrode and the results show two reversible redox peaks correspondin g probably two electrons from Mo centers.Visible absorption spectrosc opy shows three absorption bands corresponding to the pπ–dπ charge transfer transitions of O → Mo bonds. However, single crystal X-ray diffraction analysis reveals that thecompound contains an anionic cluster β-octamolybdate (β-$[Mo_8O_{28}]^{4-}$), stabilized by the cation (counterion) dihexyammonium $[CH_3\ (CH_2)_5\text{-}NH_2^+\text{-}(CH_2)_5\text{-}CH_3]$. In addition, XRD shows the existence of two types of hydrogen bonds in the compound: single hydrogen bonds (N – H---O) and bifurcated hydrogen bonds (N – H---(O, O). Therefore, these Hydrogen bond interactions ensure the cohesion of the structure into a three-dimensional network.

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Introduction:-

Polyoxometalates (POMs) are clusters of metal oxides resulting from an interconnection of $[MOx]^{n-}$ polyhedra where M is a metal center generally belonging to VB or VIB group of transition metals. In the majority of cases, the environment around the metallic element M (Mo, V, W, etc.) is generally octahedral, tetrahedral rarely trigonal bipyramidal, whose peripheral atoms are oxygen atoms and often its degree highest oxidation rate is Mo(IV), V(V), W(VI) [1]. Indeed, POMs, as a type of important metal oxide clusters, have been used as inorganic building blocks for constructing supramolecular networks with various organic compounds [2-3].

However, a single cluster can give rise to the existence of a large number of rigid structures and stable structural isomers. Since some years, the POMs compounds, in particularly polyoxomolybdates clusters, have attracted great attention due to their remarkable properties and their structural varieties, impacting several fields of application as catalysis, electrical conductivity, magnetism, optical properties, medicine, biology, electrochemistry [4-9]. In previous work carried out in our laboratory by Sarr and al.[10] that the β-octamolybdate cluster can be stabilized by dialkylammonium counterions and in 2020, B. Sarr and al.,[11] reported two new inorganic-organic hybrid materials based on β and γ-octamolybdate clusters.

Given that octamolybdates are a subclass of the POM family which has been widely studied thanks to their diverse structures as well as their interesting properties. Currently, nine isomeric forms of octamolybdates have been prepared, namely a, b, g, d, ε, z, x, h and q isomers [12-14]. And these isomers are distinguished by the number of tetrahedral (MoO_4), bipyramidal (MoO_5) or octrahedral (MoO_6) units contained in their structures. Furthermore, isomerization has already been identified theoretically and experimentally in classical phases of the Keggin, Dawson, Anderson, Lindqvist type and in many other clusters [7, 15].

Continuing our research in this family of compounds [10-11, 16]. We synthesized a new compound $(C_{12}H_{28}N)_4[Mo_8O_{28}].3H_2O$ (**1**) containing a β-octamolybdate through the introduction of dihexylamine (CH_3-$(CH_2)_5$-NH-$(CH_2)_5$-CH_3). Dihexylammonium [CH_3-$(CH_2)_5$-NH_2^+-$(CH_2)_5$-CH_3] was chosen for the stabilization of the β-octamolybdate isomer [16]. The compound was characterized by spectroscopic studies (FTIR and UV-Visible) then by X-ray diffraction and by electrochemical properties

Experimental Section: -

Materials:-

Dihexylammonium (99%), iodic acid (99%) and ammonium heptamolybdate tetrahydrate (100%) were purchased from Sigma-Aldrich and used without further purification. Distilled water was used as solvent.

Synthesis: -

The compound was obtained from a one-pot synthesis, by dissolving ammonium heptamolybdate tetrahydrate (2 g, 1.72 mmol), dihexylamine (1.275 g, 6.88 mmol), and iodic acid (0.604 g, 3.43 mmol) in 50 ml of water. The solution obtained was heated to reflux and stirred for two hours. The solution was cooled to room temperature and filtered. After five days of evaporation in an oven at 323 K, a few white single crystals were obtained.

Spectroscopy measurements: -

The UV-Visible spectrum was recorded in the 200-1000 nm region with a speed of 600 nm/min and with strong smoothing using the Evolution 300 UV-VIS device controlled by VISIONpro software at room temperature in aqueous solution at Cheikh Anta Diop University in Dakar (Senegal). 0.15 mg of the compound was solubilized in 10 ml of distilled water and the distilled water was used as a blank.

Infrared spectra was recorded from a Perkin Elmer FTIR spectrometer in the 4000-400 cm^{-1} region at Cheikh Anta Diop University in Dakar (Senegal). The UV-Visible spectra were recorded in methanol in the region 200-1000 nm with a speed of 600 nm/min and with strong smoothing using the Evolution 300 UV-VIS device controlled by VISIONpro software using Cheikh Anta Diop University of Dakar (Senegal).

Table 1: Main absorption bands in the IR of the compound $(C_{12}H_{28}N)_4[Mo_8O_{28}].3H_2O$.

Frequency (cm-1)	Assignment
3500-3400	Stretching vibration of H_2O
1645 ; 1451	Deformation vibrations of H_2O
3152	Stretching vibrations of N–H
2900-2670	Stretching vibrations of C-H
872	Stretching vibrations of Mo–Ot
717 ; 469	Stretching vibrations of ν(Mo–O–Mo) and ν(Mo-(μ-O))

Electrochemical measurements: -

The electrochemical proprieties of compound were investigated in aqueous acid medium H_2SO_4 (1M) with a scan rate of 40 mV/s.

Structure determination: -

Single-crystal X-ray diffraction data collection of the compound was performed on a Oxford Diffraction XCalibur-2 with graphite monochromated Mo-Kα radiation (λ = 0.71073 A°) at 298 K. Data collection reduction and multiscan ABSPACK correction were performed with CrysAlisPro (Rigaku Oxford Diffraction). The structure was solved by direct method using SIR-92 and refined with SHELXL 2015 [16-17]. Crystallographic information files were compiled with WinGX[18]. The crystallographic data of the compound was summarized in Table 2.

Table 2: Crystallographic data of the compound $(C_{12}H_{28}N)_4[Mo_8O_{28}].3H_2O$.

Empirical formula	$C_{48}H_{112}Mo_8N_4O_{31}$
Formula weight	2008.93
Temperature/K	298.15
Crystal system	monoclinic
Space group	$P2_1/m$
a/Å	12.937(3)
b/Å	23.995(4)
c/Å	17.654(4)
α/°	90
β/°	103.53(2)
γ/°	90
Volume/Å^3	5328(2)
Z	2
ρ_{calc}g/cm^3	1.252
μ/mm^{-1}	0.965
F(000)	2024.0
Crystal size/mm^3	0.24 × 0.09 × 0.06
Radiation	MoKα (λ = 0.71073)
2Θ range for data collection/°	3.924 to 52.738
Index ranges	$-14 \le h \le 9$, $-19 \le k \le 29$, $-21 \le l \le 13$
Reflections collected	10745
Independent reflections	7111 [R_{int} = 0.2036, R_{sigma} = 0.5031]
Data/restraints/parameters	7111/395/276
Goodness-of-fit on F^2	0.992
Final R indexes [I>=2σ (I)]	R_1 = 0.1623, wR_2 = 0.3986
Final R indexes [all data]	R_1 = 0.4461, wR_2 = 0.5705
Largest diff. peak/hole / e Å^{-3}	1.21/-0.72
CCDC number	2333663

Results and Discussion: -

Fourier transform infrared (FTIR) spectroscopy: -

In the IR spectrum of (**1**), we note broad bands around 3500 cm^{-1} whose correspondence can be attributed to the νOH and νNH stretching vibration of water molecules and dihexylammonium cation $[C_{12}H_{28}N]^+$ and those which appear between 1620 and 1550 cm^{-1} are characteristic of deformation vibrations δ(O-H) and δ(N-H). The bands from 2900 to 2670 cm^{-1} can be attributed to the stretching vibrations of C-H bonds, while that from 1164 cm^{-1} at 1079 cm^{-1} are a strain vibration δ(C–H). The two bands at 940 and 875 cm^{-1} can be attributed to ν(Mo–Ot) stretching vibrations while those from 830 to 599 cm^{-1} are attributed to ν(Mo–O–Mo) and ν(Mo-(μ-O)) vibrations [19-20].

UV-Visible spectroscopy: -

The UV-Visible absorption of the compound (**1**) has been analyzed in the range 200-1000 nm in aqueous solution. This spectrum reveals three absorption peaks at 216, 231 and 255 nm. These absorption bands are attributed to the pπ–dπ charge transfer transitions of the O → Mo bonds [21-22].

Electrochemical properties: -

The redox properties of the compound were studied by cyclic voltammetry in H_2SO_4 (1M) using glassy carbon as a reference electrode. The voltammogram of the compound in the range -1250 mV to 1750 mV is reported in fig. However, we observe two reversible redox peaks and the average peak potentials $E_{1/2}$ = (Epa + Epc)/2 are 568 mV (I-I') and 183 mV (II-II') (scan rate: 40 mV/s) probably corresponding to the two consecutive two-electron reduction processes of the Mo centers. These reduction processes are similar to those reported by (L. Yaffa and al., 2020)[22]for the compound $(C_5H_{16}N_2)_3[P_2Mo_5O_{23}].4H_2O$ and (Zhang, X and al., 2016)[19]for the compound $(NH_4)_4[(NH_3\text{-}CH_2\text{-}CO)_2(Mo_8O_{28})].2H_2O$.

Structure description and discussion: -

The asymmetric unit of the compound shown in (Figure 1) consists of an anion $[Mo_8O_{28}]^{4-}$ two dihexylammonium cations $[C_{12}H_{28}N]^+$ and three water molecules. Indeed, one of $[C_{12}H_{28}N]^+$ has an occupancy factor of 1/2 because they are each in the vicinity of a center of symmetry at (1/2, 1/2, 0). On average, in the crystal, it has two symmetrical orientations with respect to these centers. This is why the unit of given form is $(C_{12}H_{28}N)_4[Mo_8O_{28}].3H_2O$ (we take two asymmetric units to have integer stoichiometric coefficients). The structure of the compound consists of interactions between the anions $[Mo_8O_{28}]^{4-}$octamolybdate, and the cations $[C_{12}H_{28}N]^+$. Each anion of octamolybdate interacts with the hydrogen atoms of the nitrogen (N_1 and N_2) of the dihexylammonium cations. In these interactions, there are two types of hydrogen bonds: single hydrogen bonds (N – H---O) and bifurcated hydrogen bonds (N – H---(O, O) (Figure 2 and 3 in green line).

In addition to the electrostatic attractions between the ions in the structure, hydrogen bonds are mainly responsible for the stability of the crystal. Additionally, these hydrogen bond interactions ensure the cohesion of the structure into a three-dimensional networkThe compound $(C_{12}H_{28}N)_4[Mo_8O_{28}].3H_2O$ is characterized by the presence of $[Mo_8O_{24}]^{4-}$octamolybdate anion where all molybdenum atoms are in their highest oxidation state Mo(IV). The complete octamolybdate anion is generated by a crystallographic inversion center. The $[Mo_8O_{28}]^{4-}$ cluster consists of eight $[MoO_6]$ octahedra sharing distorted edges, as clearly shown in figure 4b. In the cluster, all Mo atoms are bonded to two terminal oxygen atoms except Mo1 which is bonded to only one oxygen atom (O1) and Mo4 which is bonded to three oxygen atoms terminals (O4A ; O4B and O4W) see (Figure 4a).

The oxygen cluster sites appear into four categories: terminal oxygen atoms (Ot: O1, O2A, O2B, O3A, O3B, O4A, O4B and O4W), oxygen atoms connecting two molybdenum atoms (μ2 -O: O12, O23 and O34), oxygen atoms connecting three molybdenum atoms (μ3-O: O124 and O134) and oxygen atoms connecting four molybdenum atoms (μ4-O: O123). The Mo-O bond lengths can be formally divided into three groups: short bonds between [1.649 - 1.749 Å] which are linked the molybdenum atoms and the terminal oxygen atoms (Mo-Ot) in exception of Mo4-O4W (1.985 Å), average bonds between [1.918 – 2.228 Å] in which the oxygen atoms are connecting two or three molybdenum atoms (μ2-O and μ3-O) and long bonds ranging of [2.244 – 2.386 Å] in which oxygen atoms are connecting four molybdenum atoms (μ4-O: O123) see Table 3. The O–Mo–O angles values between 69.4 (8) to 176 (1) °. Details of the compound's binding geometry are summarized inTable 3. All values presented are in good agreement with data for similar structures [23-26].

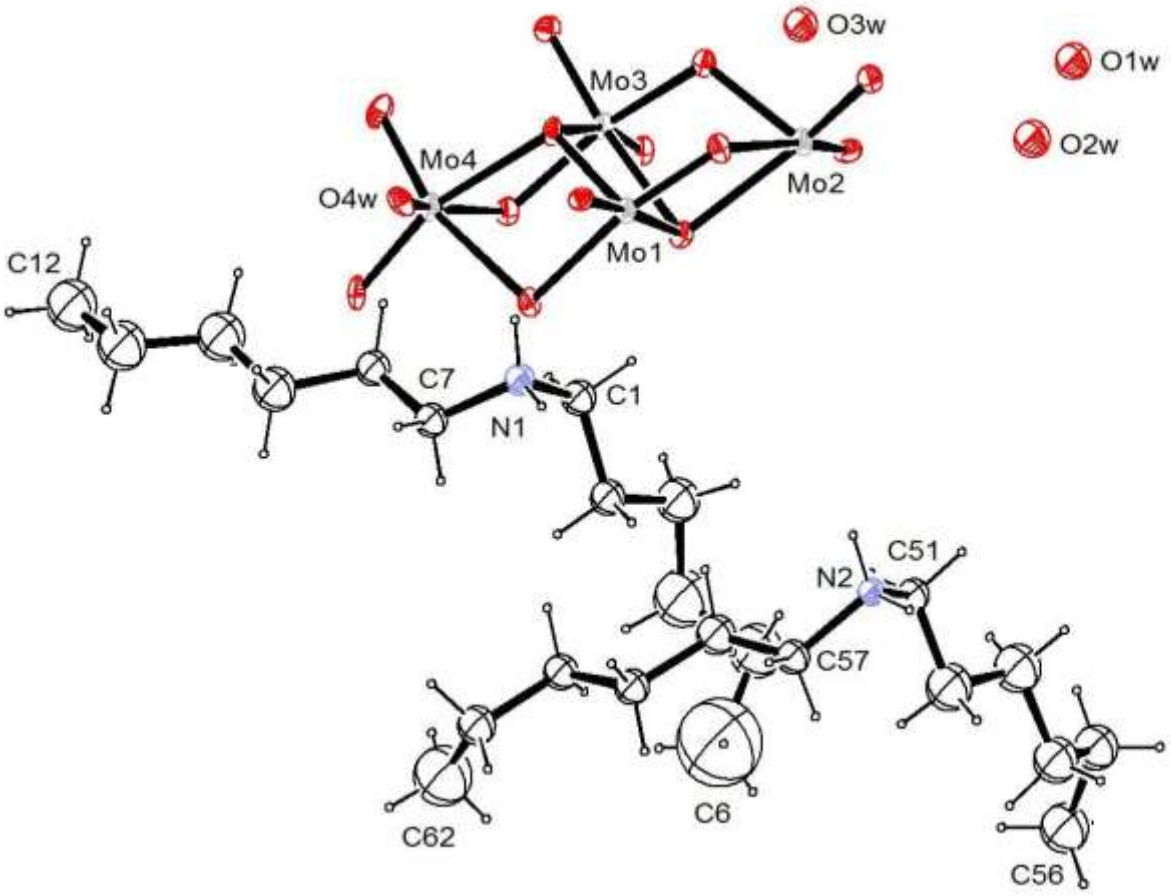


Figure 1: The asymmetric unit of the compound with displacementellipsoids drawn at the 50% probability level

Table 3: Mo-O bond measurements

Type de liaison Mo-O	Mo-Ot	Mo-O (μ2-O)	Mo-O (μ3-O)	Mo-O (μ4-O)
Mo1-O(Å)	Mo1-O1(1.694)	Mo1-O12(1.692)	Mo1-O124(2.149) Mo1-O134(1.919)	Mo1-O123(1.919) Mo1-O123(2.386)
Mo2-O(Å)	Mo2-O2A(1.649) Mo2-O2B(1.658)	Mo2-O12(2.383) Mo2-O23(1.959)	Mo1-O124(1.918)	Mo2-O123(2.301)
Mo3-O(Å)	Mo3-O3A(1.687) Mo3-O3B(1.661)	Mo3-O34(1.923) Mo3-O23(1.947)	Mo3-O134(2.298)	Mo3-O123(2.244)
Mo4-O(Å)	Mo4-O4A(1.645) Mo4-O4B(1.749) Mo4-O4W(1.985)	Mo4-O34(2.192)	Mo4-O134(2.228) Mo4-O124(2.192)	

The overall structure of the compound can be described as a superposition of infinite layers interconnected via hydrogen bonds (Figures 2 and 3).

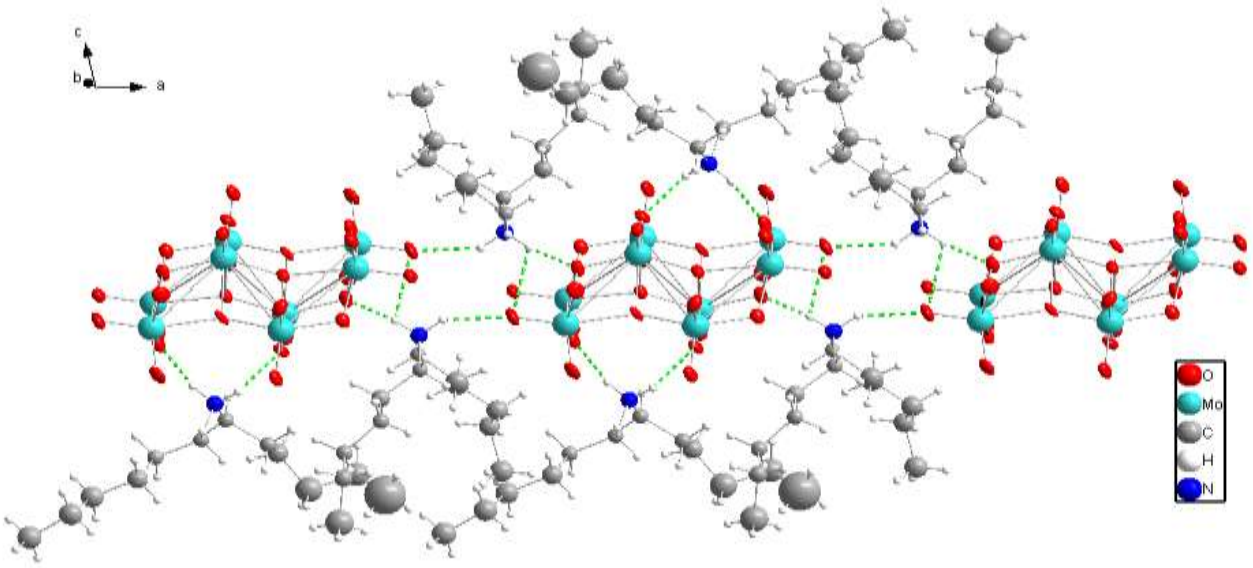


Figure 2:Detail of the interconnections of the anions $[Mo_8O_{28}]^{4-}$ with the cations $[C_{12}H_{28}N]^{4+}$ showing the hydrogen bonds (in green line)

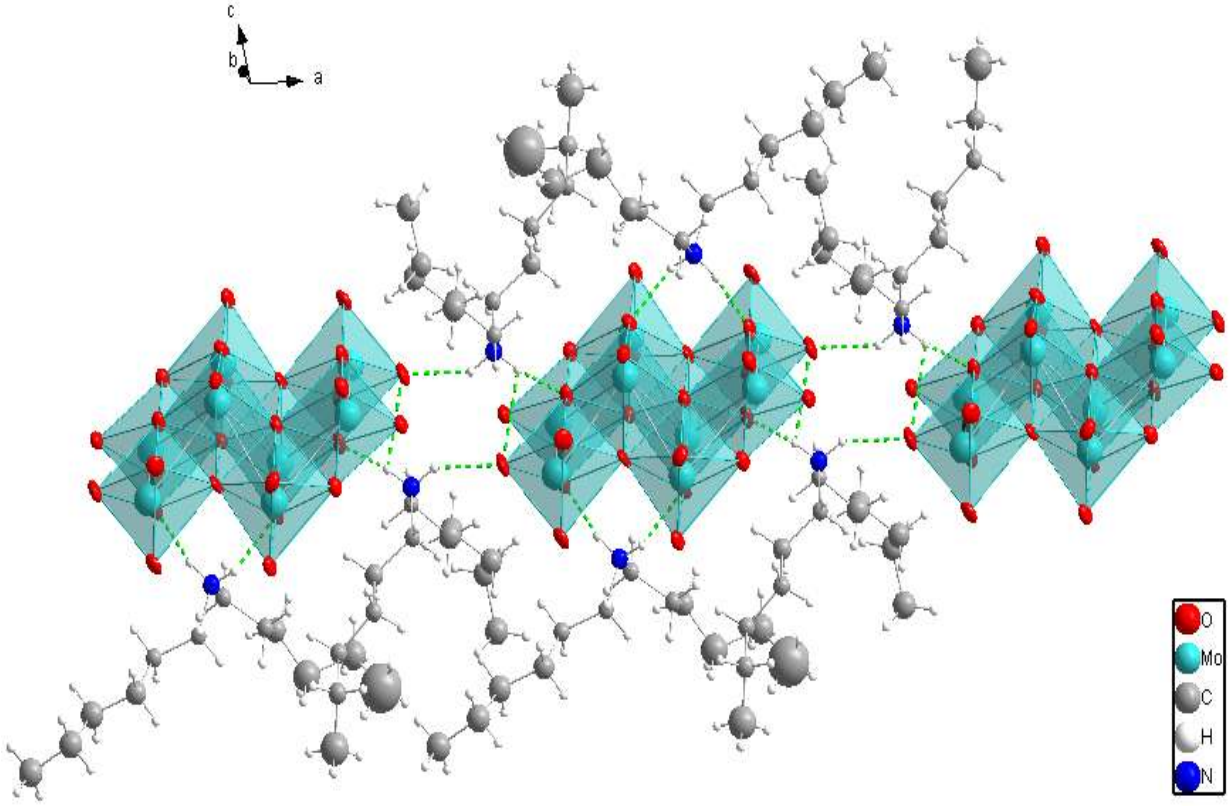
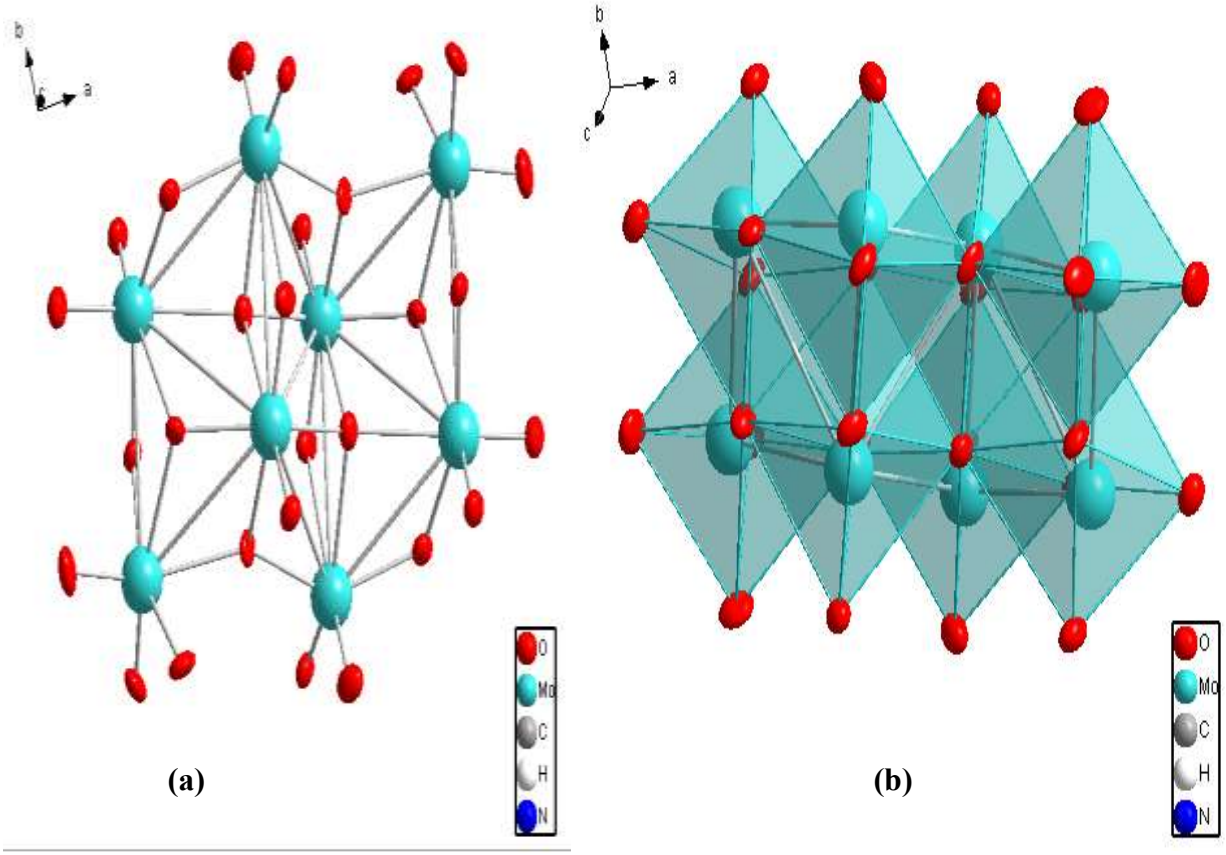


Figure 3: Detail of the interconnections of the anions $[Mo_8O_{28}]^{4-}$ represented in polyhedral form with the cations $[C_{12}H_{28}N]^{4+}$ showing the hydrogen bonds NH---O (in green line)

Figure 4 : Structures éclatées (a) polyédriques (b) de l'anion $[Mo_8O_{28}]^{4-}$octamolybdate



In annexe of the document, we have presented the IR spectrum (in Figure 5), the UV-visible spectrum (in Figure 6) and the cyclic voltammograms (in Figure 7) of $(C_{12}H_{28}N)_4[Mo_8O_{28}].3H_2O$

Conclusion:-

In summary, this new inorganic-organic hybrid material containing an isomer of the β-octamolybdate anion stabilized by the dihexylammonium cation has been successfully isolated and well characterized by spectroscopic studies (FTIR and UV-Visible), by electrochemical studies. but also by X-ray diffraction.However, the electrochemical properties of the compound showed two consecutive reversible redox processes corresponding to two electrons from the Mo centers.

And from X-ray diffraction, we noticed that the stabilizing entity (dihexylammonium) having several potential hydrogen bond donor sites connects to octamolybdate anions (nucleophiles), via single hydrogen bonds (N – H-- -O) and bifurcated (N – H---(O, O), to form thermally stable 2D or 3D supramolecular structures. In the future, we plan to extend this work to other new inorganic-organic hybrid materials in order to research their photochemical properties in the solid state and to improve their electrochemical properties.

Annex:

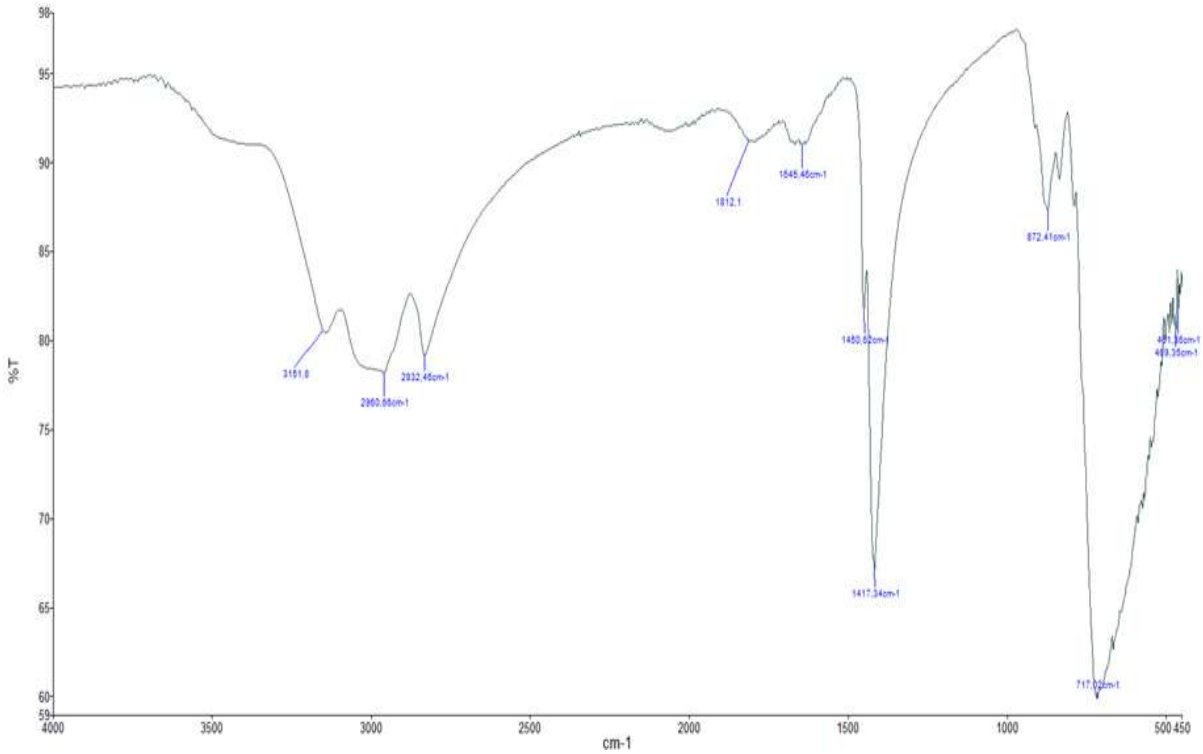


Figure 5 : FT-IR spectrum of $(C_{12}H_{28}N)_4[Mo_8O_{28}].3H_2O$

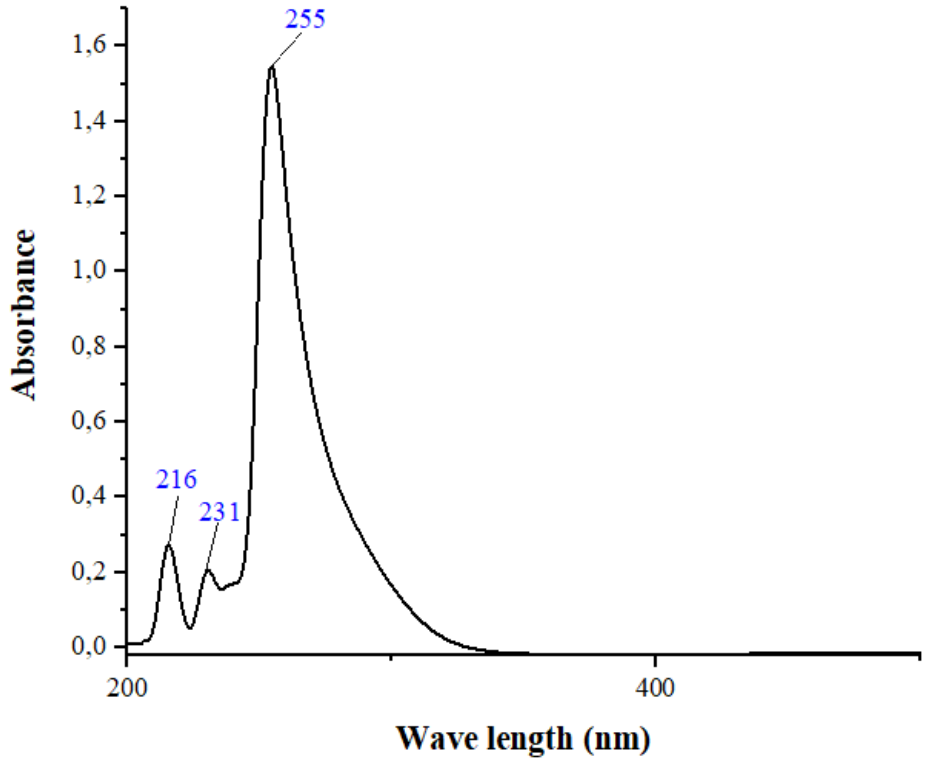


Figure 6: UV-Vis absorption spectrum in Distilled Water of $(C_{12}H_{28}N)_4[Mo_8O_{28}].3H_2O$

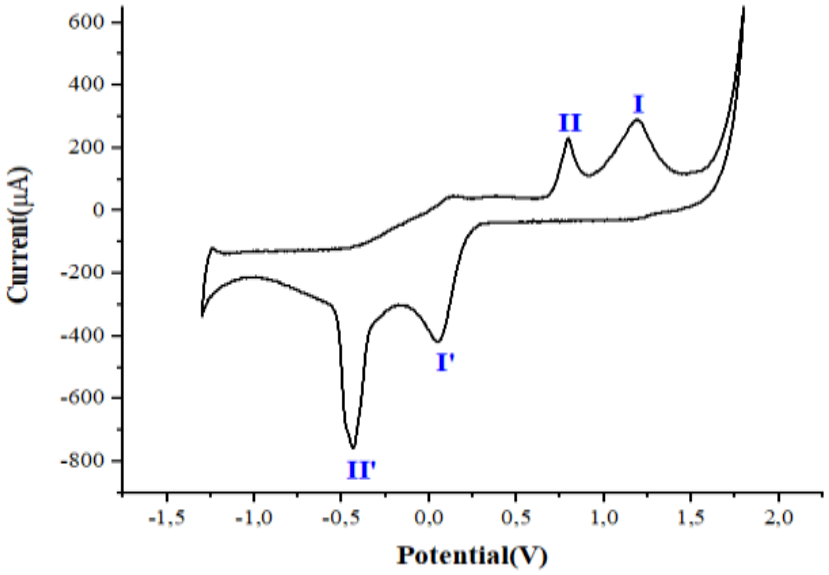


Figure 7:Cyclic voltammograms of the 1 CPE in H2SO4(1M) at scan rate =40 mV/s of compound (C12H28N) 4[Mo8O28].3H2O

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