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COMPARITIVE STUDIES OF KEGGIN & SILVERTON POLYOXOMETALATE BASED COMPLEXES AND ITS BACTRIAL STUDIES

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Abstract

Comparative studies on Silverton type and keggin doped polyoxometalate with organic compound 3-(Aminopropyl)-Imidazole (3-API) hybrid materials were prepared by using hydrothermal method and characterized by FTIR, UV-Visible Spectroscopy, XRD, and application studies Anti-microbial. The band at 210 nm with band gap 5.8 are corresponding to the POM-1(keggin type). and 209 nm 5.9 eV band gap attributed to the PMO-2 (Silverton type anion), both the polyoxometalates with the organic compound exhibits approximately similar average crystallite size 56.85 nm and for POM-1and 56.57 nm. Gram negative Salmonella and gram-positive Enterococcus are 10±1 and 10±2, respectively, according to the POM-1 result. Gram positive (Enterococcus) and gram negative (Salmonella) are 10±1 and 10±3, respectively, in POM-2. Bacterial cell walls and produced complexes have been well-interacted to provide efficient and effective bacterial activity.

Keywords: Polyoxometalates (POM), Silverton, Keggin, (3-API), Gram positive (Enterococcus), gram negative (Salmonella), Anti-Microbial.

1. Introduction

More than one compound combines together to form the hybrid material. In this hybrid materials know a day the polyoxometalates (POMs) act as a important role. There are numerous applications for polyoxometalates (POMs), which are intricate transition metal-oxygen clusters with unique topologies and multiple functions [1]. For its discoverer, the ammonium phosphomolybdate with the $[PMo_{12}O_{40}]^{3-}$ ion is commonly referred to as the Keggin structure [2]. POMs are renowned for their distinct chemical and physical characteristics, which include strong acidity, redox activity, and good chemical and thermal stability. POMs are used in many different areas, including materials research, energy storage, and catalysis. [3,4]. Their diverse spectrum of chemical and structural characteristics makes them valuable in a number of fields, including materials research, energy storage, and catalysis [5,6]. Keggin-type POMs are one of their many configurations that have been created specifically to catalyze hydrogen storage devices. Their ability to absorb and indicate electrons during the process is made feasible by their numerous redox-active metal cores and bridging oxygen atoms [7]. Because of their remarkable chemical and structural diversity, redox potential, surface charge, thermal stability, and changeable acidity,
polyoxometalates (POMs) have garnered a lot of attention lately in the field of hydrogen storages [8]. POMs are a family of inorganic compounds made up of metal-oxo clusters, which usually comprise oxygen atoms and transition metals. A few typical POM structures include Keggin $[\{XM_{12}O_{40}\}^{3/4-}]$.(X=P, Si, B and M=and M=Mo, W)[9].

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The Keggin structure is a version of the polyoxometalate (POM) known as the Dawson structure. $[X_2M_{18}O_{62}]$ n- [10,11]. In contrast, hydrothermal synthesis uses a water-based solution as the reaction medium to create materials or compounds at high temperatures and high pressures. Typically, hydrothermal synthesis goes through the following phases [12]. The potential uses of polyoxometalates (POMs) in medicine, such as drug administration, imaging, and cancer treatment, have been investigated. [13]. Researchers have looked into the possible antibacterial properties of polyoxometalates (POMs). The type of ligands, the metal ion selection, and the molecule's general structure can all affect the production of POMs and their antibacterial efficacy. It has been demonstrated that POMs exhibit antibacterial action against both Gram-positive and Gram-negative bacteria [14]. By altering the molecule's structure, for as by changing the metal ions or ligands, POM's antibacterial activity can be maximized. The concentration of POMs and the duration of bacterial contact can also affect the antibacterial action. Numerous techniques, including the disk diffusion method and minimum inhibitory concentration, can be used to assess the antibacterial activity of POMs [15]. $C_7H_{12}N_2$ is the chemical formula for the organic compound 3-(aminopropyl)-imidazole. It is derived from imidazole, a heterocyclic molecule with five members that has two nitrogen atoms in its ring. There are numerous uses for 3-(aminopropyl)-imidazole in biochemistry and chemical synthesis [16]. It is commonly used as a ligand in coordination chemistry, interacting with metal ions to generate coordination complexes. These substances can be used as catalysts in a variety of reactions, such as hydrogenation, oxidation, and cross-coupling. This effort has addressed the production and characterization of polyoxometalate of the Silverton and Keggin types. According to antibacterial research, the region of inhibition indicates that the targeted microorganism's efficacy against pathogenic bacteria increases after 180 minutes of exposure to visible light [17].

Polyoxometalates (POMs) are a fascinating class of inorganic compounds comprised of transition metal-oxygen clusters with unique topologies and multifunctional characteristics [1]. A well-known example is the ammonium phosphomolybdate with the $[PMo_{12}O_{40}]^{3-}$ ion, commonly referred to as the Keggin structure [2]. POMs are distinguished by their robust acidity, redox properties, and high chemical and thermal stability, making them useful across fields such as materials science, energy storage, and catalysis [3,4]. Their rich chemical and structural diversity enhances their potential in these areas [5,6].

Among the various POM configurations, Keggin-type POMs have been specially designed for applications in hydrogen storage, capitalizing on their redox-active metal centers and bridging oxygen atoms to facilitate electron absorption and transfer during reactions [7]. Owing to their exceptional redox potential, tunable acidity, thermal stability, and structural diversity, POMs are gaining prominence in hydrogen storage technologies [8]. In general, POMs consist of metal-oxo clusters formed from oxygen atoms and transition metals, with Keggin structures represented by the formula $[\{XM_{12}O_{40}\}^{3/4-}]$ (X = P, Si, B; M = Mo, W) [9]. Another significant POM structure is the Dawson structure, represented by $[X_2M_{18}O_{62}]n-$ [10,11].

Hydrothermal synthesis—a process that employs aqueous solutions under elevated temperature and pressure is a widely used method to create these and related compounds [12]. Beyond catalysis and materials science, POMs are also being investigated for biomedical applications, including drug delivery, imaging, and cancer treatment [13]. Researchers have explored their antibacterial activity, which depends on factors such as the type of ligands, metal ion composition, and structural configuration. POMs have demonstrated antibacterial effects against both Gram-positive and Gram-negative bacteria [14], and structural modifications, such as changes in metal ions or ligands, can further enhance this activity. Additionally, antibacterial efficacy is influenced by

POM concentration and bacterial exposure time, and is typically measured using methods like disk diffusion and minimum inhibitory concentration tests [15].

The organic compound 3-(aminopropyl)-imidazole ($C_7H_{12}N_2$), a derivative of imidazole with two nitrogen atoms in its five-membered ring, plays a significant role in this context [16]. It is widely used as a ligand in coordination chemistry to form metal-ligand complexes, which serve as catalysts in reactions including hydrogenation, oxidation, and cross-coupling.

In this work, both Keggin- and Silverton-type POMs were synthesized and characterized. Antibacterial studies revealed that, following 180 minutes of visible light exposure, these complexes significantly improved the inhibition zones of targeted pathogenic bacteria, highlighting their potential for antimicrobial applications [17].

2 Experimental

2.1 Chemicals used:

All the chemicals and reagents were purchased from Merck 3-(aminopropyl)-imidazole, Ammonium molybdate, di-sodium hydrogen phosphate (Na_2HPO_4), Sulfuric acid (H_2SO_4)]. All the chemicals were used without further purification.

2.2 Preparation of polyoxometalate (POM) based complexes:

2.2.1 Preparation of Silverton type polyoxometalate

The Silverton type polyoxometalate is prepared by dissolving ammonium-di molybdate (1.30 g; 2 mmol) and ammonium ceric sulfate (1 g; 1 millimole) was dissolved in 30 ml of deionized water and mixed under constant stirring for 10 -15 minutes at 63°C. Then, (3-Aminopropyl)-imidazole (3-API) (1 g; 0.5 millimole) was dissolved in 10 ml of deionized water. To this content, sulfuric acid is added drop by drop with continuous stirring for 20 -40 minutes. The solution was transferred into 100ml of stainless Teflon auto-clave at100° for 2–4 hours. Then the solution was kept for cooling down to room temperature. The blue color precipitate was obtained, filtered and dried at room temperature through slow evaporation [18].

2.2.2 Preparation of Keggin type polyoxometalate

The Keggin type polyoxometalate is prepared by dissolving ammonium-di molybdate (1.30 g; 2mmol) and di-sodium hydrogen phosphate (1.0 g; 1.0 millimole) was dissolved in 30ml of deionized water and mixed under constant stirring for 10 -15 minutes at 63°C. Then, (3-aminopropyl)-imidazole (3-API) (1g; 0.5millimole) was dissolved in 10 ml of deionized water. To this content, sulfuric acid is added drop by drop with continuous stirring for 20 -40 minutes. The solution was transferred into 100ml of stainless Teflon auto-clave at 100° for 2-4 hours. Then the solution was kept for cooling down room temperature. The precipitate was obtained in yellow color; The precipitate was filtered and dried at 32° c (room temperature) through slow evaporation [18,19].

3 Results and Discussion

3.1 UV-Visible Spectroscopy:

At 200-800 nm radiation range is absorbed. The observed bandfor the polyoxometalate using UV-visible spectrum in the range of 200-800 nm. Transition band is observed and band gap are calculated are given in Table1. The UV-visible band of two type polyoxometalate is shown in the figure.3.1 and 3.2.The band at 210 nm and 5.8 eV band gap are corresponding to the POM-1(kegging type). The band 209 nm and 5.9 eV band gap are attributed to the PMO-2 (Silverton type anion), Due to the inter- valence charge transfer the formation of - API with polyoxometalate is formed[5,6,7].

Table 1. UV-Visible data of POM -1, POM -2.

Complexes	Absorption band (nm)	Band gap (eV)
POM -1	210	5.87
POM -2	209	5.93

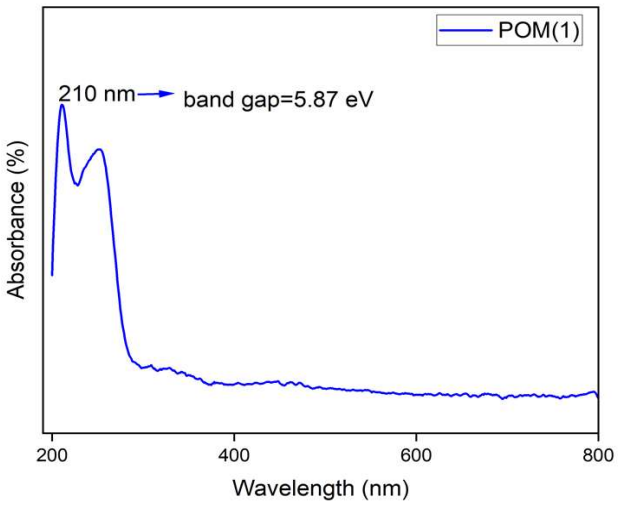


Figure -3.1 UV-Vis spectrum POM-1

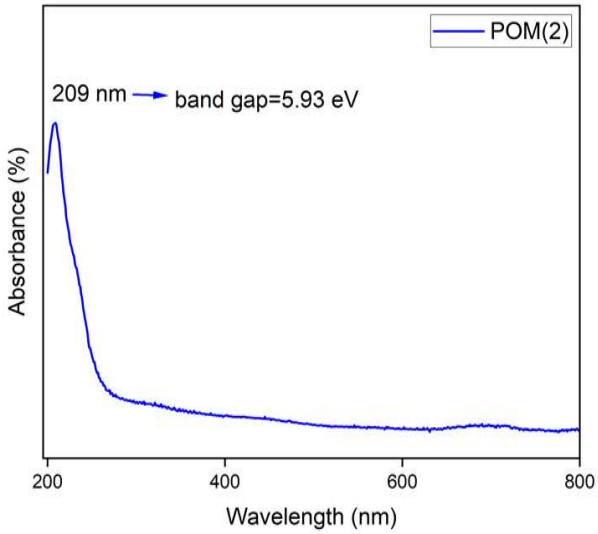


Figure-3.2 UV-Vis spectrum POM-2

3.2. FT-IR Analysis:

3.2.1 FT-IR spectrum of POM -1

FT-IR spectra of POM-1 is shown in figure3.3. Ammonium dimolybdate is shown on the finger print region of POM-1 peaks of 1200-600 cm^{-1}. ThePOM peak at 1106 cm^{-1} shows that vibration ($M-O_t$) stretching band . A peak at 883 cm^{-1} to 1106 cm^{-1} correspond to vibration ($M-O_b$) stretching bands. The POM-1 peak 618 cm^{-1} is attributed to vibration (M-O-M). The absorption of organic molecule API, the 3140 cm^{-1} peaks belongs to N-H stretching bond. The POM 1421 cm^{-1}peak vibration is corresponds to the (N-H) group. At 1597 cm^{-1} indicates aromatic C-H functional group. At 1578 cm^{-1}peak exhibits the presence (C=N) ring & peak 478 cm^{-1} is assigned to the (M-O) bond. The POM-1 absorbed peaks are corresponded to the fingerprint region of the prepared POM-1 complex [7,19]. The FT-IR spectra of POM-2 is given in table.2.

Table 2. FT-IR spectral data of POM-1

Complexes	POM (cm^{-1})				API(cm^{-1})			
	$M-O_t$	$M-O_b$	M-O-M	M-O	N-H bond	N-H stretching/ C=N ring		C-H aromatic
POM-1	1106	883-953	618	478	3140	1421	1578	1597

3.2.1 FT-IR spectrum of POM -2:

The FT-IR spectra of POM-2 is shown in figure3.4. The peak belongs to POM-2 at 888 cm^{-1}- 1050 cm^{-1} indicates vibration ($M-O_t$) stretching band . A peak at 826 cm^{-1}- 925 cm^{-1} correspond to vibration ($M-O_b$) stretching bands. ThePOM-2 peak at 619 cm^{-1} attributed to vibration (M-O-M). The absorption of organic molecule API, one at 3124 cm^{-1} shows N-H bond. Around 1454 cm^{-1}- 1575 cm^{-1} peak vibration are corresponding to the of (N-H) group. At 1610 cm^{-1} indicates to the aromatic C-H functional groups. The peak appears at 1454 cm $^{-1}$ and 1575 cm^{-1}exhibit the presence (C=N) ring & the peak 485 $cm^{-1}$is attributed to the M-O stretching band. The absorbed peak are corresponding to the fingerprint region of the prepared POM-2 complex. The FT-IR spectra of POM-2 is given in table.3.

Table 3. FT-IR spectral data of POM-2

Complexes	POM (cm^{-1})				APIcm^{-1}			
	$M-O_t$	$M-O_b$	M-O-M	M-O	N-H bond	N-H stretching/ Bending	C=N ring	C-H aromatic
POM-2	1050	836-925	619	485	3124	1575	1454	1610

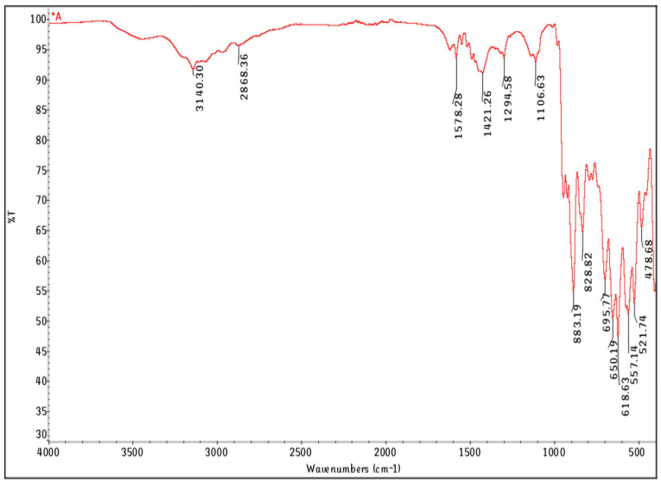


Figure -3.3 : FT-IR spectrum of POM-1

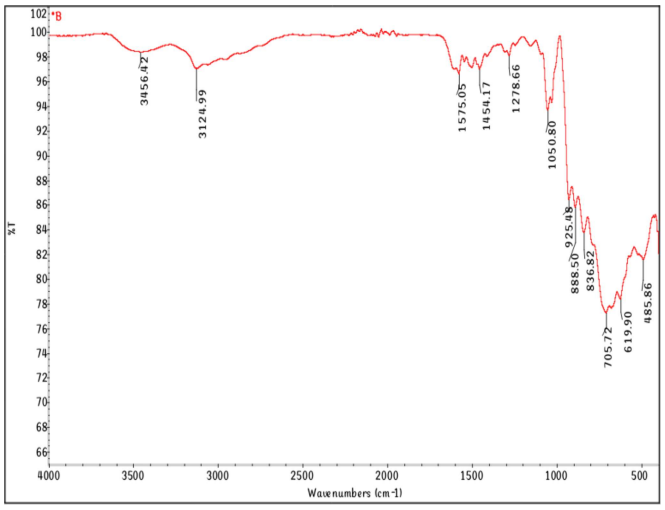


Figure 3.4: FT-IR spectrum of POM-2

3.3 XRD

The XRD pattern of POM-1, POM-2 are shown in. The grain size of the prepared compound is given in table. The characteristics peaks also API. The crystalline size is estimated according to the Debey - Scherer equation,

$$D = k\lambda / \beta\cos\theta$$

The crystalline size of the prepared POM is around 40-80 nm approximately, the average crystallite size of the Keggin doped POM 56.85 and 56.57 for silverton based POM [20,21]. The peak of POM-1 is at 9.74°,1059°,12.10°,13.21°,23.55°,25.59°,26.39°,30.87° are corresponds to the POM-1 which confirms the prepared complex is shown in figure.3.3.1.The peak of POM-2 is at 9.38°, 15.38°, 17.35°, 26.16°, 29.09°, 49.82°, 73.47° are corresponds to the POM-2 confirms the prepared complex is shown in figure.3.3.2.

Table. 4 XRD patterns of POM-1, POM-2:

S.no	Complex	Theta (2θ)	Grain Size (nm)
1	**POM-1**	*9.74*	*58*
		10.59	*40*
		12.10	*48*
		13.21	*63*
		23.55	*72*
		25.59	*52*
		26.39	*65*
2	**POM-2**	*9.38*	*55*
		15 37	*50*
		17.38	*49*
		26.16	*51*
		29.09	*52*
		49.82	*64*
		73.47	*75*

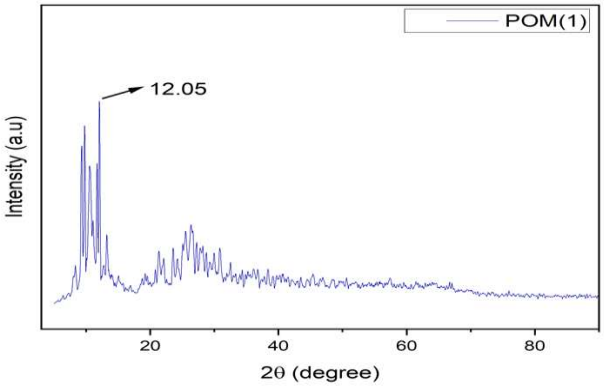


Figure 3.4 XRD patterns of POM-1

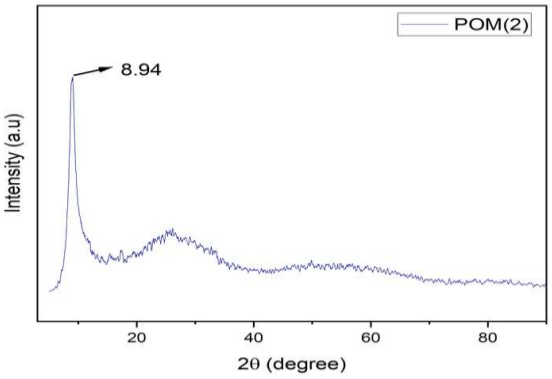


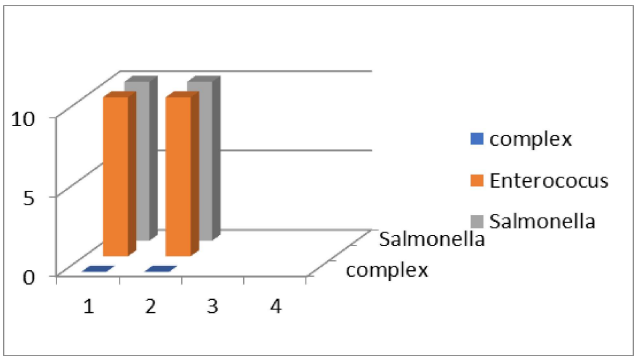
Figure 3.6 XRD patterns of POM-2

3.4 Anti-microbial activity

The Antimicrobial studies of the POM-1 and POM-2 are tested in vitro by the well distributed method. The bacteria of Enterococcus and Salmonella has been used to finding antimicrobial property of different POM's. The zone inhibition values for POM -1, POM -2 and control values shows increased in activity [22,23]. The Antimicrobial studies of the compound acts on gram positive and gram-negative cell activity. The result for POM-1 shows gram positive (Enterococcus) 10±1, and 10±2 gram negative (Salmonella) [24]. POM-2 shows gram positive (Enterococcus) 10±1, and 10±3 gram negative (Salmonella) shown in figure 3.5 and 3.6. The cell wall of the bacterial has been well interact along with prepared complexes to make the bacterial efficient and effective activity [25,26].

Table.5. Anti-microbial activity of POM-1, POM-2:

Complex	Enterococcus (mm)	Salmonella (mm)
POM-1	10±1	10±2
POM-2	10±2	10±3



Bar diagram of Anti-microbial activity of POM1, POM2

Fig 3.5 The (Gram^{+ve})E.coccus & (Gram+ve) salmonellaanti-microbial activity for POM-1

Fig 3.6 The (Gram $^{+ve}$) E.coccus & (Gram $^{-ve}$) Salmonella anti-microbial activity for POM-2

4 Conclusions

The complexes of POM-1 and POM-2 have been synthesized and characterised using spectral techniques such as UV-Spectroscopy, FTIR spectroscopy, X-ray diffraction (XRD), Antimicrobial study. The IR spectral data shows the characteristic peaks which confirm the formation POM complexes. The finger print region for the POM'S have been observed. The crystalline size of the synthesized complexes is calculated at around 40-80nm. The POM complexes show good anti-bacterial activity of Gram positive and Negative of about 10±1 mm and 10±2mm for the POM-1 complex and 10±2 mm and 10±3mm for the POM-2 complex. However, POM-2 (Silverton type) shows slightly better antibacterial activity (especially against Gram-negative bacteria) compared to POM-1 (Keggin type).

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Conflict of Interest: Nil

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