

Ion Exchange Behaviour of Newly Synthesized Pb²⁺ Selective Cerium (IV) Iodomolybdate Ion-Exchanger and Catalytic Activity in Dye Removal

Saly Jaber¹, N. Kohila^{2,*}, Fatima Hamouche³, Mika Sillanpää⁴, Fatma Bassyouni⁵, Luigi Orlando Freire Martínez⁶

¹Department of Analytical Chemistry, Saint Joseph University, Beirut, Lebanon.

²Department of Chemistry, Aditanar College of Arts and Science, Affiliated to Manonmaniam Sundaranar University, Tirunelveli, Tamil Nadu, India.

³Department of Chemistry, Ibn Tofail University, Kenitra, Rabat-Salé-Kénitra, Morocco.

⁴Department of Chemical Engineering Technology, University of Johannesburg, Johannesburg, Gauteng, South Africa.

⁵Department of Chemistry of Natural and Microbial Products, National Research Centre, Cairo, Egypt.

⁶School of Engineering and Applied Sciences, Technical University of Cotopaxi, Latacunga, Cotopaxi, Ecuador.
saly.jaber@usj.edu.lb¹, nkohila302@gmail.com², fatima.hamouche@uit.ac.ma³, mikas@uj.ac.za⁴, fatma.nrc@hotmail.com⁵, luigi.freire@utc.edu.ec⁶

Abstract: The main purpose of the present work is the synthesis and characterization of a new inorganic ion exchanger of a three-component system, namely cerium (IV)iodomolybdate. Cerium (IV) iodomolybdate was prepared successfully by co-precipitation using the optimum reagent ratios. The structural and morphological properties of the optimised exchanger, cerium (IV) iodomolybdate, were studied by SEM-EDS, XRD and FT-IR. To evaluate its functional value, researchers determined its cation exchange capacity by column and pH titration methods. In addition, researchers performed distribution coefficient experiments in distilled water to further validate the results. The material also showed high chemical stability in very acidic, neutral and basic environments. For practical environmental applications, researchers have explored dye uptake from wastewater, and batch testing was the method of choice for this particular topic. The researchers tested several contact times, exchanger dosages, and initial dye concentrations to determine the efficacy of the dye removal process. The results show that cerium (IV) iodomolybdate-based ion exchangers have strong ion-exchange performance and excellent thermal and chemical stability, as well as repeatability. This material is highly selective for toxic heavy metals and an excellent dye adsorbent, with tremendous potential for developing innovative technologies for environmental remediation. This is a fantastic potential.

Keywords: Cerium (IV) Iodomolybdate; Inorganic Ion-Exchanger; Co-Precipitation Method; Cation Exchange Capacity; Distribution Coefficient; Chemical Stability; Heavy Toxic Metals.

Received on: 19/10/2024, **Revised on:** 14/01/2025, **Accepted on:** 15/05/2025, **Published on:** 15/09/2026

Journal Homepage: <https://www.fmdbpublish.com/user/journals/details/FTSASS>

DOI: <https://doi.org/10.69888/FTSASS.2026.000770>

Cite as: S. Jaber, N. Kohila, F. Hamouche, M. Sillanpää, F. Bassyouni, and L. O. F. Martínez, “Ion Exchange Behaviour of Newly Synthesized Pb²⁺ Selective Cerium (IV) Iodomolybdate Ion-Exchanger and Catalytic Activity in Dye Removal,” *FMDB Transactions on Sustainable Applied Sciences*, vol. 3, no. 2, pp. 89–104, 2026.

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1. Introduction

*Corresponding author.

The contamination of water resources by heavy metals and hazardous organic pollutants has become a critical global concern [1]. To address the resulting environmental risks, significant efforts have focused on developing advanced wastewater management techniques that minimise waste generation and improve effluent quality [2]. Among current water-processing technologies, inorganic ion exchangers have gained prominence because of their superior thermal stability, chemical resistance, and radiation tolerance compared to traditional organic resins [3]. These materials, including synthetic zeolites, hydrous metal oxides, and insoluble heteropolyacid salts, are highly effective for selective heavy-metal recovery [4]. Heteropolyacid salts, particularly those derived from 12-heteropolyacids, are of particular interest because incorporating rare earth elements (like cerium) increases the material's surface acidity, significantly improving adsorption efficiency [5]. Such selective exchangers are a well-established method for separating and removing toxic cations from industrial waste [6]. Recent research has focused on synthesising multicomponent systems to overcome the limitations of simple salts [7].

Previous studies have demonstrated the efficacy of cerium-based materials: Sheha and El-Khouly [10] synthesized thermally stable cerium(IV) phosphotungstate for the quantitative separation of metal pairs such as Pb^{2+} - Hg^{2+} and Pb^{2+} - Cd^{2+} . Siji and Janardanan [11] established the role of mixed-metal exchangers in heavy metal removal and organic dye adsorption. Preetha and Janardanan [12] demonstrated the potential of cerium-based quaternary systems (e.g., CZPT) as efficient metal scavengers. Mixed or double-salt exchangers are often preferred because they offer greater structural stability, selectivity, and ion-exchange capacity than their simple counterparts [8]. Given the persistence and non-biodegradable nature of industrial dyes and the harmful impact of toxic heavy metals, there is a clear demand for novel, multifunctional materials that act as both ion exchangers and efficient catalysts for environmental remediation [14]. In this study, researchers synthesize and characterize a novel three-component inorganic ion exchanger, cerium(IV) iodomolybdate [9]. Researchers use advanced analytical techniques, including FT-IR, XRD, and SEM-EDS, to investigate its structural properties and chemical stability [15]. Furthermore, Researchers evaluate their efficacy as ion exchangers for metal recovery and as catalysts for degrading organic dyes in wastewater, providing a comprehensive assessment of their utility in hazardous waste remediation [13].

2. Literature Review

Inorganic ion exchangers have attracted significant interest compared with conventional organic ion exchange resins because of their excellent thermal stability, radiation resistance, ion selectivity and chemical durability [16]. Their ability to selectively remove toxic metal ions and radionuclides from contaminated water has made them highly valuable in environmental remediation, wastewater treatment, and nuclear waste management. Inorganic exchangers are more resistant to high temperatures, oxidizing conditions and intense radiation fields than organic resins and are therefore particularly useful in demanding industrial and radioactive waste applications. Early investigations into radioactive waste treatment emphasized that inorganic ion-exchange materials have remarkable stability and long-term performance under extreme operating conditions, establishing them as preferred materials for nuclear decontamination processes [1]. Over time, researchers have refined synthesis techniques, allowing them to tailor new multicomponent inorganic exchangers with improved selectivity and adsorption efficiency. Early developments in this field focused on heteropoly acid-based inorganic ion exchangers, which showed good cation-exchange behavior due to their porous structure and high density of exchangeable functional groups. Incorporating transition metals into phosphate, molybdate, tungstate and arsenate frameworks greatly enhanced ion-exchange capacity and chemical resistance. Janardanan and Nair [2] prepared antimony (III) arsenate and antimony (III) molybdate and showed that these materials exhibited remarkable ion-exchange characteristics, satisfactory reproducibility and chemical stability.

Their work established the importance of combining multiple inorganic constituents to improve exchanger performance. Similar investigations by Gupta et al. [4] on zirconium (IV) selenomolybdate further showed that heteropoly acid-based exchangers had a high affinity for multivalent metal ions while maintaining excellent resistance under acidic conditions. These studies provided the foundation for designing complex inorganic ion exchangers capable of efficiently separating toxic heavy metals. Growing environmental concern about contamination in industrial wastewaters has expanded the use of inorganic ion exchangers beyond radionuclide separation. Heavy metals such as lead, cadmium, mercury, copper, chromium, and nickel remain persistent environmental pollutants because they are non-biodegradable and accumulate within biological systems. Traditional treatment methods usually have low selectivity, high operating costs and secondary pollution. Thus, inorganic ion exchangers have attracted interest as a potential alternative owing to their high selectivity, easy regeneration and structural rigidity. Naushad [3] extensively reviewed inorganic and composite ion-exchange materials and their wide applications in environmental remediation, catalysis, wastewater purification and analytical chemistry. The review revealed the higher adsorption capacities of composite inorganic exchangers due to synergistic interactions between the individual components and encouraged further research on multicomponent hybrid materials. Inorganic ion exchangers based on cerium have attracted special attention because cerium has multiple oxidation states, good redox properties and good structural stability. These features can significantly improve the ion-exchange efficiency and the adsorption selectivity.

Nilchi et al. [5] studied cerium-substituted phosphate exchangers and found that including cerium significantly improved cation-exchange capacity, with remarkable thermal and chemical stability. Their research also showed improved selectivity for radioactive and toxic heavy metal ions, indicating great potential for environmental remediation and nuclear waste treatment. Later studies on cerium (IV) molybdate, Nilchi et al. [16], further confirmed that cerium-based exchangers have high exchange capacities, excellent mechanical strength and efficient ion-separation capabilities. These results strongly support further development of cerium-containing composite exchangers for advanced environmental applications. Another important step in ion-exchange technology was the development of organic–inorganic hybrid ion exchangers. Hybrid materials combine the mechanical flexibility of polymers with the thermal and chemical stability of inorganic matrices, overcoming many shortcomings of conventional exchangers. Inamuddin et al. [6] synthesised Nylon-6,6–zirconium(IV) phosphate hybrid ion exchangers, which exhibited enhanced ion-exchange capacity, mechanical strength and structural stability. FTIR, XRD and thermal analyses confirmed the successful integration of organic and inorganic phases. Similarly, Kelta et al. [13] reported new reactive functional polymer-based inorganic exchangers with superior exchange capacities and improved durability in aggressive chemical environments. These studies show how structural changes can improve exchanger performance. The synthesis methodology is a key factor in determining the physicochemical properties of inorganic ion exchangers.

Co-precipitation remains one of the most commonly used preparation techniques because of its excellent compositional homogeneity, controlled particle-size distribution, and reproducibility. Optimising reagent concentration, mixing ratios, pH, and aging conditions directly affects crystallinity, porosity, and exchange capacity. Characterization techniques such as SEM (scanning electron microscopy), EDS (energy-dispersive X-ray spectroscopy), XRD (X-ray diffraction), and FTIR (Fourier transform infrared spectroscopy) have become essential tools for confirming structural integrity and identifying the functional groups responsible for ion exchange. Several studies have shown that combining these analytical techniques provides a complete understanding of the exchanger's morphology, crystallinity, elemental composition and bonding characteristics to optimise synthesis protocols [16]; [6]. Distribution coefficient (K_d) measurements remain one of the most important methods for assessing the ion selectivity of newly synthesised exchangers. The higher the K_d value, the higher the affinity for a particular metal ion and thus the better applicability for selective separation. Yavari et al. [7] studied titanium molybdophosphate exchangers and reported high selectivity for cerium and strontium ions, suggesting their potential application in radioactive waste management. Their work showed that exchanger performance depends strongly on solution chemistry and competing ionic species. Nabi et al. [15] also synthesised titanium (IV) molybdosilicate and investigated its analytical applications through detailed studies of distribution coefficients, confirming its high selectivity and excellent reproducibility.

These studies showed that distribution coefficient analysis is an important parameter for evaluating exchanger performance. The ion-exchange efficiency is strongly affected by water hardness and competing ions in practical wastewater treatment systems. Natural waters are rich in calcium and magnesium ions, which compete with the target contaminants for adsorption sites. Vasilyuk et al. [8] investigated the Effect of water hardness on the removal of copper ions by ion-exchange-assisted electrodialysis. They found that competing hardness ions can reduce the exchange efficiency by an order of magnitude unless the exchangers are highly selective. This observation underlines the need to develop new inorganic exchangers with improved selectivity toward target pollutants in complex aqueous environments. The main performance parameters of inorganic ion exchangers in industrial applications are thermal stability and chemical resistance. In highly acidic or alkaline environments, many traditional exchangers cannot retain their structural integrity, thus limiting their practical application. Cerium-containing exchangers show good resistance across wide pH ranges because of their stable crystal structures. The materials reported by Nilchi et al. [16] and Gupta et al. [4] exhibited excellent stability in acidic, neutral, and alkaline media. They thus can be used for repeated adsorption-desorption cycles without significant degradation. This stability significantly reduces operational costs and extends the exchanger's lifetime. Several researchers have also developed molybdate-based composite exchangers for their excellent selectivity for multivalent cations. Molybdate frameworks offer plentiful active exchange sites while also providing structural rigidity.

Sheha and El-Khouly [10] reported that incorporating molybdate groups into inorganic matrices has significantly improved ion-exchange behavior. Similarly, Siji and Janardanan [11] synthesised multicomponent inorganic exchangers with higher selectivity towards heavy metals. Preetha and Janardanan [12] further showed that tuning the synthesis conditions could significantly improve exchange capacity and adsorption performance. Together, these investigations demonstrate the importance of molybdate incorporation in developing highly efficient inorganic exchangers. Considerable evolution of fundamental understanding of ion-exchange mechanisms has also resulted from classical studies on exchanger acidity and titration behaviour. Topp and Pepper [14] studied the relationships between exchanger structure and ionization characteristics through detailed analysis of titration curves. In their pioneering work, they showed that exchange capacity depends strongly on the accessibility and acidity of functional groups in exchanger frameworks. These principles are still applied to interpret the pH titration curves of newly synthesised inorganic exchangers. More recently, the environmental applications of inorganic ion exchangers have been extended to the Adsorption of synthetic dyes from industrial wastewater. The textile, pharmaceutical and chemical industries are major sources of persistent dyes, which are very harmful to the environment and health.

Inorganic exchangers, with their porous structure, high surface area and numerous exchange sites, have shown excellent dye-adsorption capacity through electrostatic interactions and ion-exchange mechanisms. Dye-removal efficiency depends on contact time, initial dye concentration, exchanger dosage, solution pH and temperature. Inorganic ion exchangers containing cerium have been observed to exhibit higher adsorption efficiency because of higher surface reactivity and more active adsorption sites Nilchi et al. [16] and Naushad [3]. The literature reports steady progress in developing high-grade inorganic ion exchangers for environmental remediation. Multicomponent materials with cerium, molybdate, phosphate, zirconium, titanium and antimony have shown excellent ion-exchange capacities, outstanding chemical stability and high selectivity to toxic heavy metals and radionuclides. However, relatively little attention has been paid to cerium (IV) iodomolybdate, especially its structural characterization, cation-exchange behavior, chemical stability and dye-adsorption efficiency. This study focuses on synthesizing and thoroughly characterizing a new cerium (IV) iodomolybdate ion exchanger prepared by co-precipitation, and on investigating its physicochemical properties and environmental remediation potential through ion-exchange and dye-removal studies. This investigation is expected to contribute to the development of efficient, stable and environmentally sustainable inorganic ion exchangers suitable for advanced wastewater treatment and heavy metal remediation technologies.

3. Experimental Section

3.1. Chemicals and Reagents

The main reagents used for the synthesis of the cerium (IV) iodomolybdate (CeIMo) ion-exchanger were cerium(IV) ammonium nitrate GR [Merck Specialties Private Limited (Mumbai)], potassium iodate [s.d. FINE-CHEM Limited (Mumbai)], sodium molybdate dihydrate, AR [Himedia Laboratories Pvt. Ltd. (Cochin)]. All other chemicals and reagents used were of analytical grade. 0.05 M solutions of cerium (IV) ammonium nitrate, potassium iodate, and sodium molybdate were prepared in demineralised water.

3.2. Instruments

pH was measured using a Systronics pH meter. A Systronics spectrophotometer-166 was used to determine optical density. 'REMI Rotatory shaker' was used for shaking the solutions for different studies. 'Shimadzu Electronic Balance' was used for weighing. All glassware used in this work was of 'Borosil' make. A glass column was used for column operations.

3.3. Synthesis of Cerium (IV) Iodomolybdate Ion-Exchanger (CeIMo)

Seven samples of cerium (IV) iodomolybdate were prepared by adding a mixture of 0.05 M potassium iodate and sodium molybdate solutions to 0.05 M cerium (IV) ammonium nitrate solution under continuous stirring, using different volume ratios. Adjust the desired pH by adding dilute HNO₃. The lemon-yellow precipitates obtained were aged in the mother liquor for 24 hours at room temperature. The precipitates of seven different samples were filtered. After filtration, all seven samples were washed continuously with distilled water to remove excess acid. The pH of the effluents was checked using pH paper. When the effluent became neutral, the precipitates were considered free of excess acid. These seven samples were then dried in an oven at 40 °C. The precipitate color was recorded (Table 1).

Table 1: Conditions for synthesis and properties of CeIMo

No.	Sample No.	Mixing volume ratio			pH Value	Appearance of precipitate
		CAN 0.05 (M)	PI 0.05 (M)	SM 0.05 (M)		
1	CeIMo-I	1	1	1	2	Lemon yellow
2	CeIMo-II	1	1	2	2	Lemon yellow
3	CeIMo-III	1	2	1	2	Lemon yellow
4	CeIMo-IV	2	1	1	2	Lemon yellow
5	CeIMo-V	2	2	1	2	Lemon yellow
6	CeIMo-VI	2	1	2	2	Lemon yellow
7	CeIMo-VII	1	2	2	2	Lemon yellow

- CeIMo- Cerium (IV) iodomolybdate
- CAN - Cerium (IV) ammonium nitrate
- PI - Potassium iodate
- SM- Sodium molybdate

For granulation, the materials were cracked and immersed in hot water. The granules from seven samples were then ready for generation. Convert the granules to H⁺ form by treating them with 1 M HNO₃ solution. For this, keep the granules from the seven samples separately in acid solution in seven different conical flasks. Repeat the process several times with fresh acid solution each time. After decanting the granules, remove the acid and keep the granules in fresh acid solution for 24 hours. After 24 hours, decant the acid and filter the granules. The granules were finally washed with distilled water to remove excess acid and dried in an oven at 40±1 °C.

3.4. Determination of Ion-Exchange Capacity (IEC)

The ion-exchange capacity of all seven samples was determined using the standard column method (Table 2). 0.5g (dry mass) of cerium (IV) iodomolybdate in H⁺ form was packed in a glass column having a glass wool support at the base. 0.1 M NaNO₃ solution was passed through the column slowly by adjusting the effluent rate at 9 – 10 drops per minute. The effluent was carefully collected in a 250 ml conical flask. The complete replacement of H⁺ form from the ion-exchanger by Na⁺ was checked by comparing the pH of the eluent (0.1 M NaNO₃) and the effluent with the help of pH paper. The collected effluents were titrated against a standard NaOH solution. Then the exchange capacity in meq/g was calculated using the formula: $IEC = \frac{av}{w}$. Where 'a' is the molarity, 'v' is the volume of alkali used during titration, and 'w' is the weight of the exchanger taken. Sample (CeIMo-III) was selected for further studies on account of its higher ion-exchange capacity in comparison to others. The sample was synthesised in bulk to support detailed study.

Table 2: Determination of ion-exchange capacity of different bulk samples

No.	Sample Number	COBBG	COBAG	Na ⁺ IEC (meq/g)
1	CeIMo	Yellow	Yellow	0.1683
2	CeIMo	Yellow	Yellow	0.8500
3	CeIMo	Yellow	Yellow	1.0233
4	CeIMo	Yellow	Yellow	0.2432
5	CeIMo	Yellow	Yellow	0.2600
6	CeIMo	Yellow	Yellow	0.1579
7	CeIMo	Yellow	Yellow	0.8231

- CeIMo- Cerium (IV) iodomolybdate
- COBBG- Color of beads before generation
- COBAG- Color of beads after generation
- IEC- Ion Exchange Capacity

3.5. Synthesis of the Selected Sample in Bulk for Detailed Study

Bulk synthesis was performed by mixing 0.05 M potassium iodate solution, 0.05 M sodium molybdate solution, and 0.05 M cerium(IV) ammonium nitrate solution in a 2:1:1 ratio under continuous stirring. Adjust the pH to -1 by adding dilute HNO₃. The precipitate was aged in the mother liquor for twenty-four hours at room temperature and filtered. The obtained matrix was washed with distilled water to remove excess acid and dried in an oven at 40±1 °C. After drying, the matrix was converted to granules as described earlier. After generating granules, excess acid was removed by washing with distilled water, and the acid-free granules were dried in an oven at 40±1 °C. These granules are now ready for detailed study. The ion exchange capacity was determined again by the column method using 0.1 M NaNO₃ solution.

3.6. Effect Of Size and Charge of Metal Ion on IEC

Similarly, the IEC of the Ce (IV) iodomolybdate cation exchanger was determined by the column method for different alkali and alkaline earth metals. Four equal parts (0.2 g each) of the cation exchanger were packed in four different columns, and distilled water was passed through each column to wash and settle the granules and form a bed. Four different metal-ion solutions of equimolar concentration (0.4 M) were prepared. Each column was treated with a particular salt solution to occur the ion exchange. The effluents were collected and titrated against a standard NaOH solution separately to determine the hydrogen ion concentration.

3.7. Effect of Eluent Concentration on IEC

Elution magnitude depends on eluent concentration. To find out the optimum concentration of eluent for complete elution of H⁺ ions, a fixed volume of NaCl solution with concentrations ranging from 0.1 M to 0.6 M was passed through a column

containing 0.2g of the exchanger in H⁺ form with a flow rate of ($\approx 0.5 \text{ mL min}^{-1}$). The effluent was titrated against a standard solution of NaOH to determine the H⁺ ions eluted.

3.8. Elution Behavior

For this study, a 0.4 M NaCl solution of optimum concentration was passed through a column containing 0.5g of H⁺ form of Ce(IV) iodomolybdate cation exchanger at a flow rate of 0.5 mL min^{-1} to elute H⁺ ions completely. The effluent was collected in 10 mL fractions, and each 10 mL fraction was titrated against a standard alkali solution to determine the H⁺ ions eluted. This experiment determined the minimum volume needed for complete elution of H⁺ ions, which indicates the column's exchange efficiency.

3.9. Effect of Time on IEC

The optimum shaking time of the CeIMo ion-exchanger with NaCl solution for complete elution of H⁺ ions was determined. 0.2g of ion-exchanger was shaken with 50 mL of NaCl solution, and the amount of liberated H⁺ ions was titrated against the standard NaOH solution at different time intervals.

3.10. pH Titration Study

Topp and Pepper [14] method was employed for pH titration. This method determines the exchanger's ion-exchange capacity. In this method, Researchers treated 0.2g of the exchanger with 25 mL of the NaCl–NaOH solution. Researchers prepared six samples by varying the NaCl–NaOH solution ratio. Researchers maintained a final volume of 25mL to achieve an ionic strength of 0.25 M. Researchers continuously shook the samples for six hours using an electric rotary shaker, then kept them as such for 24 hours to reach equilibrium. After 24 hours, the pH of the different samples was measured using a pH meter.

3.11. Chemical Stability

To determine the extent of dissolution of the material in different chemical solvents, viz. demineralised water, mineral acids like HCl and CH₃COOH, organic solvents like acetone, ether and CCl₄ and bases like NaOH. For this study, 0.2g of material was equilibrated with 25mL of the solution of interest and then left the mixture as such for 24 hrs at room temperature. If material remained in solution, it was decanted and washed with demineralized water to remove excess acids and bases. The color and weight changes were noted. This method was used to find the stability of the inorganic cation exchanger towards different solutions. After treatment, the ion-exchange capacities of all samples were determined by the column method.

3.12. Distribution Studies

The batch process determined the distribution coefficient values for various metal ions in DMW. In this process, 0.1g of Ce(IV)IMo inorganic ion exchanger in H⁺ form was equilibrated with 20 mL of different metal salt solutions. The mixture was then shaken for six hrs at room temperature using an electric rotary shaker. Then, the solutions were kept as such for twenty-four hours at room temperature. The amount of metal ions left in the solution was determined by titration against a standard EDTA solution. The blank titration was also carried out using metal ion solutions, EDTA solution and suitable indicators. The distribution coefficient (K_d) values were calculated for metal ions using the following equation:

$$K_d = (I - F / F) \times (A/W) \quad (1)$$

Where,

- I = Burette reading for the metal ion solution before treatment with ion exchanger
- F = Burette reading for the metal ion solution after treatment with ion exchanger
- A = Volume of metal ion solution taken
- W = Weight of the ion exchanger

3.13. Recovery of Calcium from Hard Water

The water samples were collected and filtered first to remove suspended particles. The samples were then used to separate and recover the heavy metal present. CeIMo, which has greater selectivity for calcium metal ions, was packed in a column; 25 mL of the water sample was passed through the column, and the process was repeated 3-4 times using the effluent collected at the bottom. This was to maximise cation uptake. The flow rate was maintained at 0.2 mL/min . 1 M HNO₃ was used as the eluent

to elute the metal ions completely from the cation exchanger column, and the metal ions were then determined titrimetrically with standard EDTA solution.

3.14. CeIMo as an Efficient Dye Adsorbent

The removal of Methyl Violet (MV) and Malachite Green (MG) dyes was studied in the presence of the CeIMo ion exchanger. Researchers prepared 50 ppm solutions of MV & MG dyes in deionized water and diluted them as needed. Researchers observed catalytic degradation when they added 100 mg of each material to the dye solution at room temperature. To determine the percentage of dye removal, Researchers collected samples at regular intervals and centrifuged them to remove fine particles. The sample absorbance at λ_{max} was measured using a UV-Vis spectrophotometer as reported earlier. Dye removal efficiency was determined using Beer's law by knowing the absorbance of the initial dye solutions (A_o) and after a specific time (A_t):

$$\text{Dye removal percentage (\%)} = [1 - (C_t / C_o)] = [1 - (A_t / A_o)] \quad (2)$$

4. Results and Discussions

4.1. Characterisation of CeIMo Ion-Exchanger

4.1.1. FT-IR Spectral Studies

Fourier transform infrared (FT-IR) spectra of CeIMo samples were recorded using a KBr pellet medium and used to identify functional groups characteristic of hetero polyacid salts. FT-IR spectrum of the sample in the region 1200 – 600 cm^{-1} is of interest, as it explains the metal-oxygen bonding in the hetero polyacid compounds (Table 3).

Table 3: Assignment for FT-IR absorption bands of CeIMo

Frequency (cm^{-1})	Assignment
3415.31	Symmetric and asymmetric –OH stretching vibrations
1628.59	Hydrogen-bonded –OH bending (H–O–H bending of adsorbed water)
1387.53	Deformation vibration of metal hydroxyl (M–OH) group
735.71	Mo–O stretching vibrations
546.72	M–O stretching vibrations

The FT-IR spectrum of CeIMo is shown in Figure 1. The FT-IR spectrum of CeIMo shows a broad band at 3415.31 cm^{-1} , attributed to symmetric and asymmetric –OH stretching, while the band at 1628.59 cm^{-1} is due to H-O-H bending.

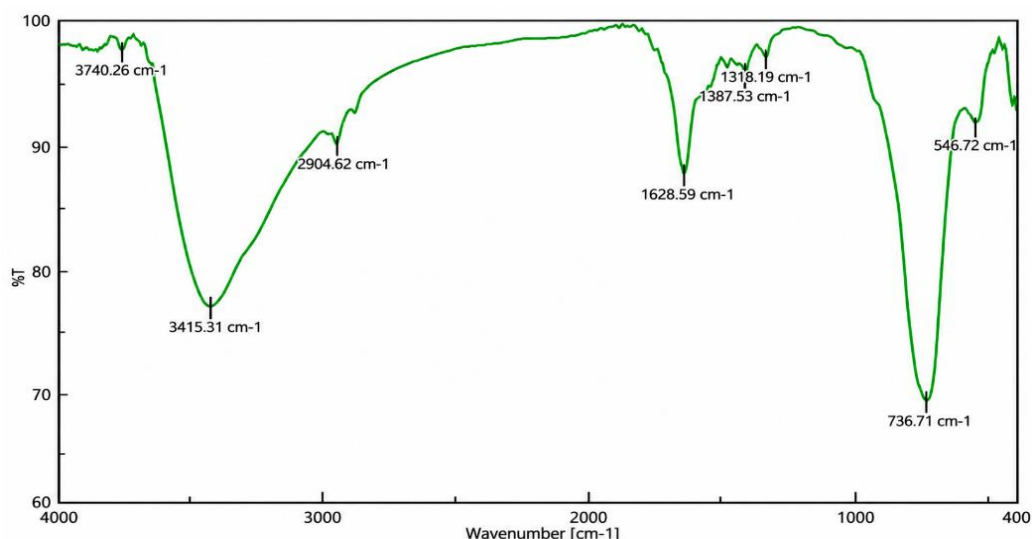


Figure 1: FT-IR spectrum of CeIMo ion-exchanger

The sharp band at 1387.53 cm^{-1} is due to deformation vibrations of metal hydroxyl groups and interstitial water. Sharp peaks at 735.71 & 546.7 cm^{-1} are attributed to metal-oxygen vibrations.

4.1.2. X-Ray Diffraction Study

X-ray analysis provides information on crystalline/amorphous content and crystallite size/orientation. X-ray diffraction studies of the CeIMo ion exchanger were done by the powder method. From XRD, particle size can be calculated using the Debye-Scherrer formula: $D = 0.96\lambda / \beta \cos \theta$. Where D is the particle size of the sample (nm), λ is the wavelength of the X-ray (1.54056 Å), β is the full width at half maximum, and θ is the diffraction angle between the incident and diffraction beam (o).

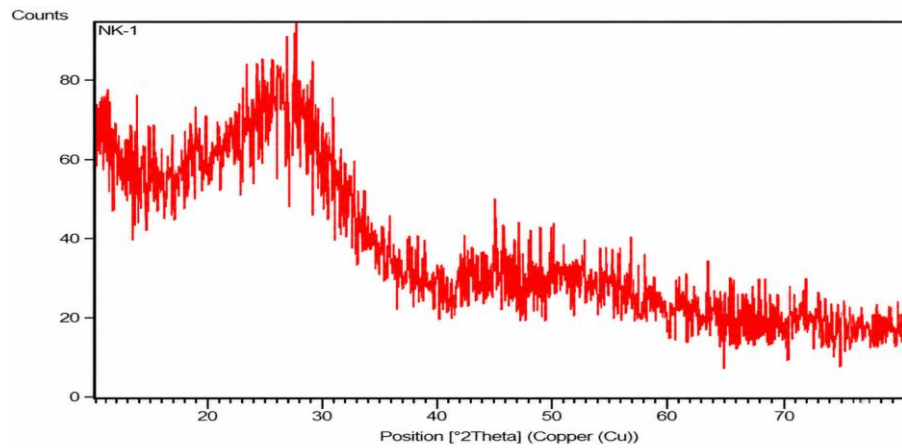


Figure 2: XRD pattern of CeIMo ion-exchanger

The X-ray diffraction pattern of cerium (IV) iodomolybdate (Figure 2) does not show sharp peaks. This indicates that the H⁺-form exchanger is amorphous.

4.1.3. SEM and EDS Analysis

SEM images reveal information about the sample's texture and orientation. Results of SEM images of cerium(IV) iodomolybdate are shown in Figure 3.

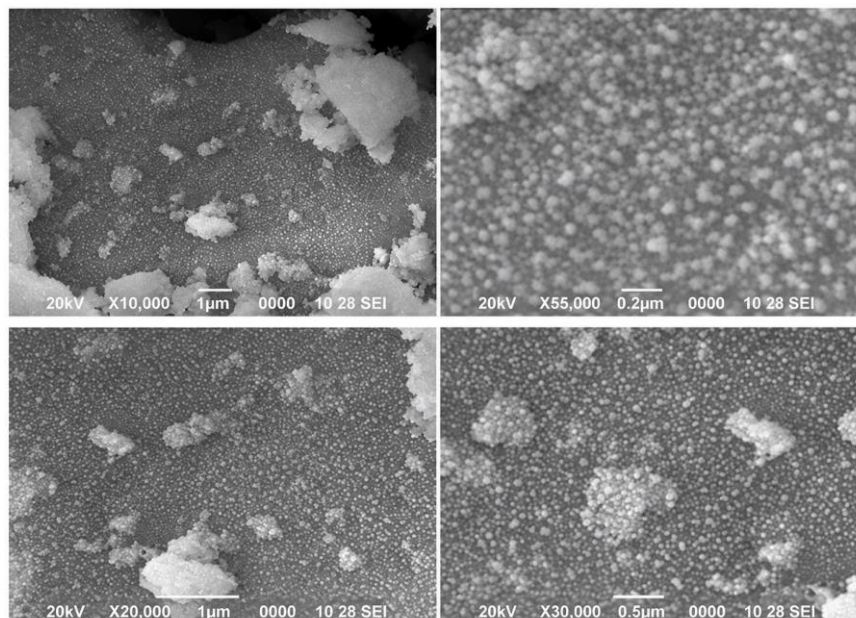


Figure 3: SEM photographs of CeIMo at different magnifications

SEM images of the Ce(IV) iodomolybdate ion exchanger show irregular particles with no sign of crystalline structure, which strongly supports the material's amorphous nature. Energy-dispersive spectrometry (EDS) was used to identify the elements in

the prepared sample, Ce(IV) iodomolybdate, by selecting a portion of an SEM image and recording the corresponding spectral peaks.

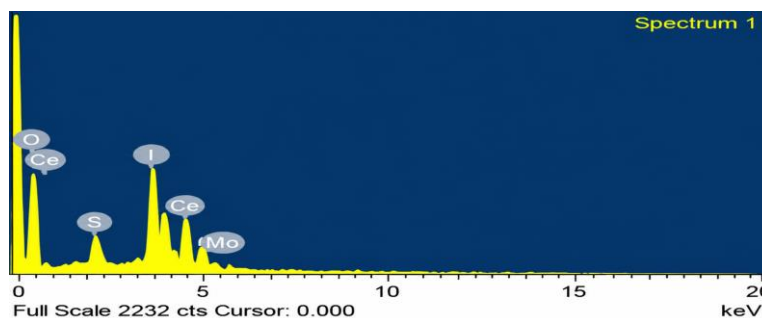


Figure 4: EDS spectrum of CeIMo ion-exchanger

The EDS spectrum of the CeIMo ion exchanger (Figure 4) shows peaks for Ce, I, Mo and O, but not for other impurities in the sample. The Weight % of Ce is 27.6, and the % of I, Mo & O were 40.1, 14.2 & 16.1, respectively.

4.2. Ion Exchange Studies of CeIMo Ion-Exchanger

The IEC of the material was determined by the column method. The Effect of the size and charge of the exchanging ion on the ion-exchange capacity was also observed for the CeIMo ion-exchange material. Table 4 shows that IEC for alkali and alkaline earth metal ions (15-16) increases with an increasing charge on the exchanging cation and an increasing atomic number (decreasing hydrated ionic radii) of the exchanging cation. Ions with smaller hydrated radii enter the cation exchanger pores more easily, resulting in higher Adsorption. The maximum ion-exchange capacity sequence for alkali metal ions is $K^+ > Na^+$, while for alkaline earth metals it is $Ba^{2+} > Sr^{2+}$. The size and charge of the exchanging ion affect the IEC of the Ce(IV) iodomolybdate cation exchanger.

Table 4: IEC of CeIMo for some monovalent and bivalent cations

Exchanging Cation	Salt Used	Ionic Radius (Å)	Hydrated Ionic Radius (Å)	IEC (meq/g)
Na^+	NaCl	0.93	2.69	0.6625
K^+	KCl	1.33	2.42	0.7125
Sr^{2+}	$SrCl_2$	1.18	5.80	0.7525
Ba^{2+}	$BaCl_2$	1.40	5.21	0.8875

- IEC - Ion Exchange Capacity
- CeIMo – Ce(IV) iodomolybdate

The optimum eluent concentration for complete elution of H^+ ions was 0.4 M NaCl, as shown in Figure 5.

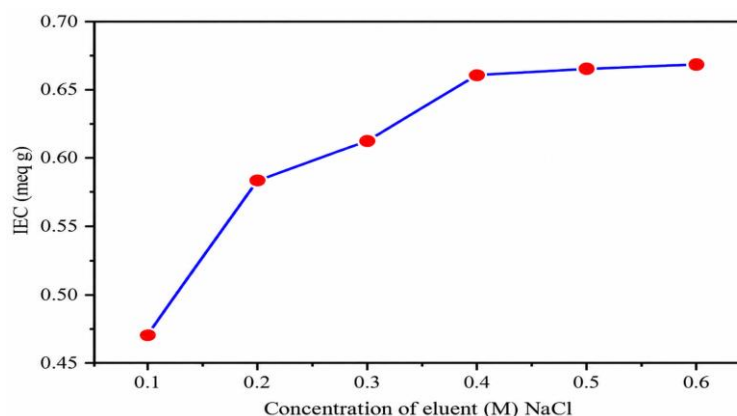


Figure 5: Exchange capacity of CeIMo as a function of eluent concentration

The elution behavior of the CeIMo cation exchanger (Figure 6) indicated that the exchange is quite fast because only 120 ml of NaCl solution is enough to release H⁺ from 0.5g of CeIMo ion exchange material. The Effect of elution time on the exchange capacity of the CeIMo ion exchanger (Figure 7) shows a constant capacity only after 90 minutes of equilibrium.

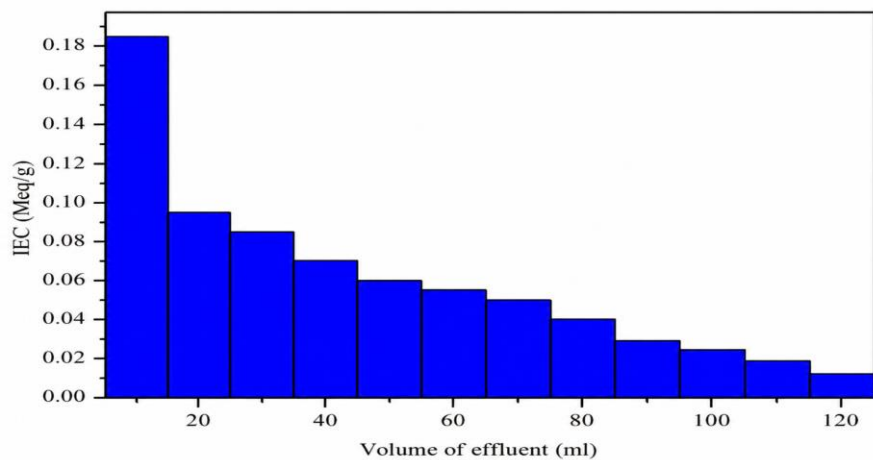


Figure 6: Elution behavior of CeIMo ion exchanger

The pH titration curve was obtained under equilibrium conditions for the NaCl –NaOH system. The pH titration curve revealed that the material behaves as a strong cation exchanger, as indicated by the low pH (1.96) of the solution when no OH⁻ ions were added to the system.

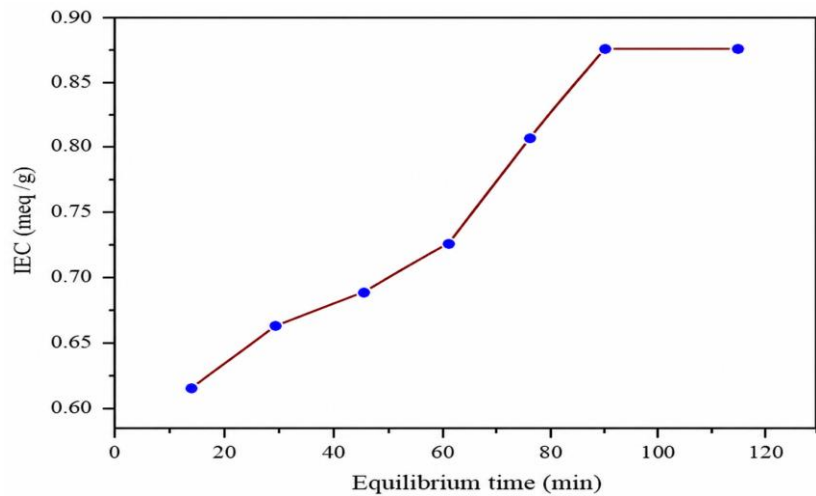


Figure 7: Effect of time on IEC of CeIMo ion exchanger

As the volume of NaOH added increases, more OH⁻ ions are consumed, suggesting an increase in the rate of ion exchange in the basic medium due to removal of H⁺ ions from the external solution (Table 5).

Table 5: pH titration values of CeIMo ion exchanger

Sample Number	NaCl – NaOH system		pH value
	NaCl Solution (ml)	NaOH Solution (ml)	
CeIMo -III	25	0	1.96
CeIMo –III	20	5	5.87
CeIMo –III	15	10	8.57
CeIMo –III	10	15	9.52
CeIMo –III	5	20	9.96
CeIMo –III	0	25	10.73

The pH titration curve of CeIMo (Figure 8) shows one inflection, indicating that the exchanger is monofunctional; two proton-binding sites are expected from the anionic groups associated with the matrix.

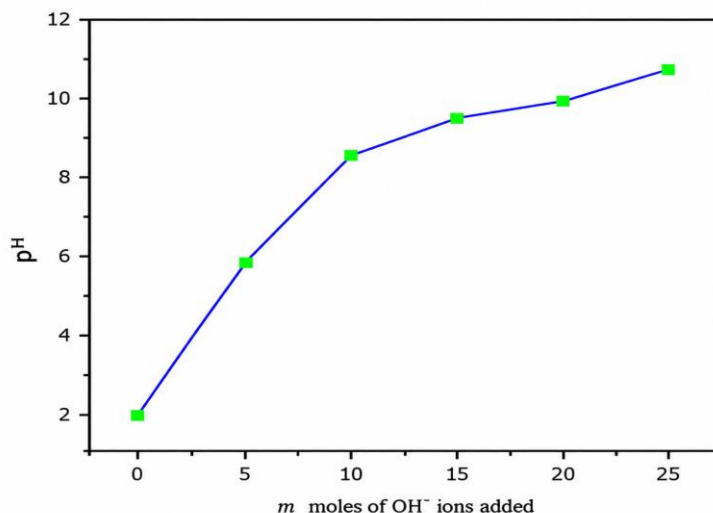


Figure 8: pH titration curve of CeIMo ion exchanger

- WI = Initial weight of exchanger before dipping in solution
- WF = Final weight of exchanger after dipping in solution

Chemical stability studies showed that the Ce(IV) iodomolybdate cation exchanger was stable in DMW, partly soluble in 2 M CH₃COOH, organic solvents like ether and CCl₄, and fairly soluble in 2 M HCl and acetone, as evidenced by no change in color or sample form. However, it was unstable in base, such as 2 M NaOH. The material became colorless in alkaline media due to hydrolysis of the exchanger at higher pH values (Table 6).

Table 6: Chemical stability of Ce(IV) iodomolybdate

No.	Solution	W _I (g)	W _F (g)	Colour	IEC (meq/g)
1	DMW	0.2	0.2	Lemon yellow	0.6875
2	2 M HCl	0.2	0.060	Pale yellow	0.75
3	Ether	0.2	0.140	Lemon yellow	0.05
4	Acetone	0.2	0.066	Lemon yellow	0.189
5	CCl ₄	0.2	0.142	Lemon yellow	0.05
6	2 M CH ₃ COOH	0.2	0.117	Lemon yellow	0.1923
7	2 M NaOH	0.2	0.068	Colourless	0.183

The selectivity of CeIMo was found to be in the order Pb²⁺ > Ca²⁺ > Ni²⁺ > Cu²⁺ > Mg²⁺. Distribution studies showed that the Ce(IV)IMo exchanger has a very high affinity for Pb²⁺ and Ca²⁺ ions and the lowest affinity for Ni²⁺, Cu²⁺, and Mg²⁺ (Table 7).

Table 7: Distribution coefficient (K_d) values of metal ions in DMW

Cation	Taken As	Distribution Coefficient, K _d (mL/g)
Ca ²⁺	Carbonate	126.1
Mg ²⁺	Sulphate	4.810
Ni ²⁺	Sulphate	10.71
Cu ²⁺	Sulphate	9.710
Pb ²⁺	Nitrate	153.7

Distribution studies showed that the material possessed exceptionally high K_d values for Pb²⁺ and was therefore considered highly selective for Pb²⁺. Pb(II) is a major water pollutant. Distribution coefficient (K_d) values were used to measure the

material's selectivity for lead metal ions. Selectivity determines how effectively the element of concern is separated from other elements. The uptake of Pb²⁺ is so strong that it may be separated from a large number of elements. The separation factor is the relative tendency of two ions to be adsorbed in an exchanger from solutions of equal concentration. It is used as a measure of the possibility of chromatographic separation and is also expressed as the ratio of the distribution coefficients of the eluents to be separated as:

$$\text{Separation factor } (\alpha_{AB}) = K_d(A) / K_d(B) \quad (3)$$

Where K_d (A) and K_d (B) are the distribution coefficients for the two competing species A & B in the ion exchange system (Table 8).

Table 8: Separation factors of different metal ions in DMW on CeIMo

Separation Factor (α)	DMW
$\alpha_{Pb, Ca}^{Pb}$	1.22
$\alpha_{Pb, Mg}^{Pb}$	31.95
$\alpha_{Pb, Ni}^{Pb}$	14.35
$\alpha_{Pb, Cu}^{Pb}$	15.82

Ce(IV) iodomolybdate ion exchanger is extensively used in the softening of hard water. Ce(IV) iodomolybdate ion exchanger has a high selectivity coefficient for calcium ions and is used to remove calcium from hard water (Table 9).

Table 9: Removal of Ca (II) from hard water

Metal ion	CeIMo Column		
	Calcium Ion Loaded (ppm)	Calcium ion eluted (ppm)	% of efficiency
Ca ²⁺	588	506	86.0

4.3. Dye Removal Studies on Ceimo Ion-Exchanger

4.3.1. Synthetic Wastewater Preparation

Accurately weighed about 1 g of dye and transferred it into a 1000ml standard measuring flask through a funnel. It was dissolved in a minimum amount of water, then made up to the mark. It was used as the 1000 ppm stock solution.

4.3.2. Measurement of Optical Density

An Elico model SL-164 UV-Vis Spectrophotometer was used with quartz cells of 1cm path length.

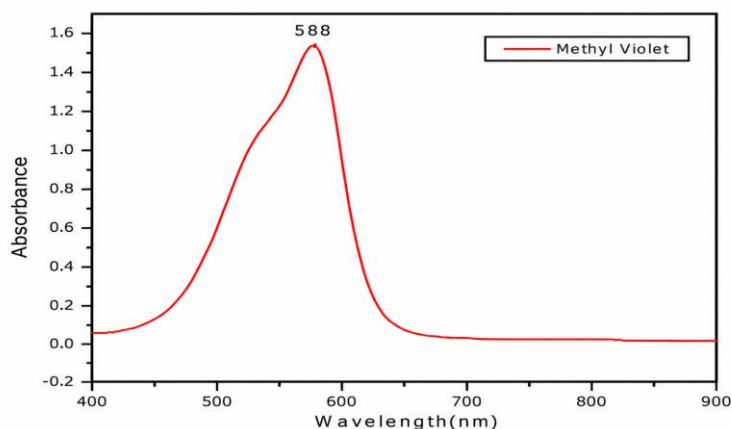


Figure 9: UV-visible spectrum of methyl violet dye

To determine the λ_{max} of the solution, a 20-ppm dye solution was placed in the quartz cell. The dye solution was scanned over the 190 –1100 nm wavelength range.

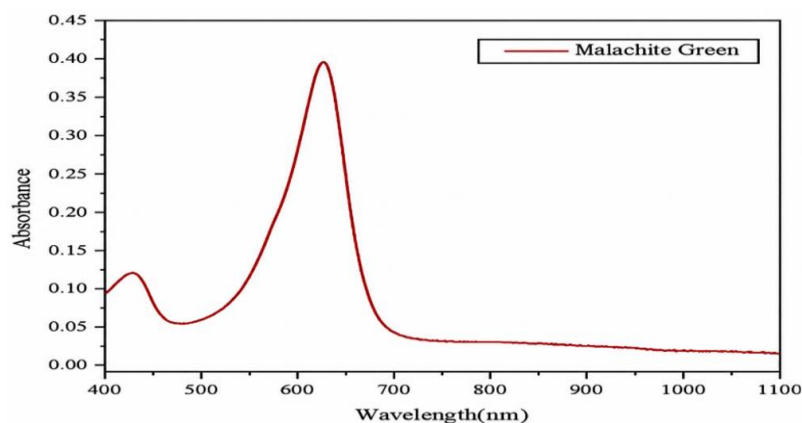


Figure 10: UV-visible spectrum of malachite green dye

The UV– Vis spectra of Methyl violet and Malachite Green solutions are given in Figures 9 and 10. λ_{max} for methyl violet and malachite green dyes were 586 nm and 619 nm, respectively.

4.3.3. Adsorption of Dyes by Batch Study

Aqueous solutions of Methyl Violet (MV) and Malachite Green (MG) (20 mL, 20 ppm) were equilibrated separately with 100 mg of exchanger (CeIMO) and were shaken for 30 minutes. The sample solutions were then filtered and centrifuged to remove the adsorbent before analysis. Absorbance was recorded before and after equilibration with a UV – Visible spectrophotometer at the absorption maximum of each dye (MV – 586 nm) and (MG – 619 nm).

4.3.3.1. Effect of Concentration on Dye Removal

The Effect of initial dye concentration on the percentage of dye removal was studied by varying the initial concentrations from 10- 40 ppm for MV and MG dyes with optimum ion exchanger loading (0.1 g).

Table 10: Effect of concentration on dye removal

Concentration of Dye (ppm)	% of Dye Removal	
	Methyl Violet	Malachite Green
10	89.9	86.4
20	89.5	83.9
30	85.7	79.0
40	75.6	78.5

Table 10 and Figure 11 show the results. Generally, the percentage of dye removal decreases as dye concentration increases.

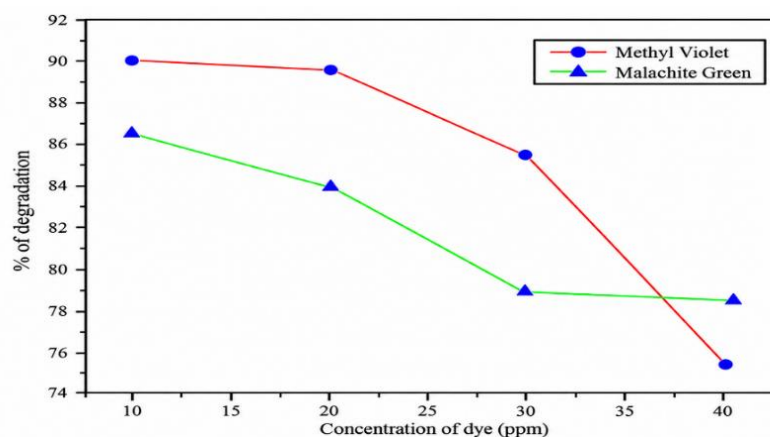


Figure 11: Plot of % dye removal vs concentration

4.3.3.2. Effect of Time Variation

The Effect of contact time on the adsorption process was studied by varying contact time and keeping the other factors constant. Table 11 shows the Effect of contact time on dye removal.

Table 11: Effect of time on dye removal

Contact time (min)	% of Degradation	
	Methyl Violet	Malachite Green
10	90.6	88.3
20	90.9	88.9
30	91.9	89.3
40	92.9	89.6

From Table 11, the % removal of MG and MV dyes is maximum at a contact time of 30 minutes. Dye removal does not increase significantly after 30 minutes (Figure 12).

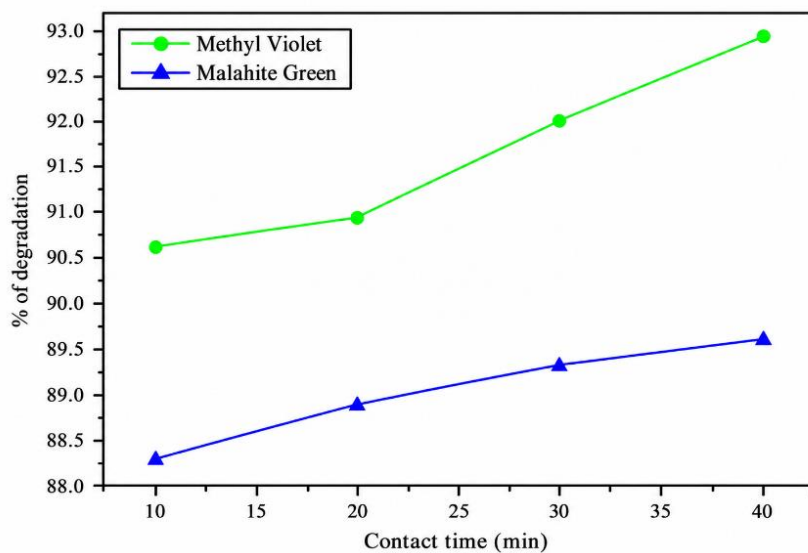


Figure 12: Plot of % dye removal vs time

4.3.3.3. Effect of Ion Exchanger Dose

The Effect of ion exchanger dose on dye removal was studied by varying the amount from 0.025, 0.050, 0.075, and 0.10g/20 mL of dye solution; the results are presented in Table 12 and Figure 13.

Table 12: Effect of dose on dye removal

Adsorbent Dose (g)	% of degradation	
	Methyl Violet	Malachite Green
0.025	76.94	71.89
0.05	90.45	77.41
0.10	90.92	83.97
0.15	91.25	88.52

The amount of ion exchanger is a key parameter in dye removal studies. To avoid using excess ion exchanger, determine the optimum loading for efficient dye removal. Table 12 shows dye removal (%) for ion exchanger amounts from 0.025g to 0.100g/20 mL of dye solution. The percentage of dye removal increases rapidly as the ion exchanger amount increases from 0.025g to 0.100g for both dyes. The optimum ion exchanger loading for dye removal was 0.1g. CeIMo removed more than 92% of Methyl Violet and 89% of Malachite Green within 30 minutes under stationary conditions. Because CeIMo contains

structural hydroxyl groups, the dyes can bind to the sorbent through hydrogen bonds or weak Van der Waals forces. Even small changes in dye structure can significantly affect adsorption capacity.

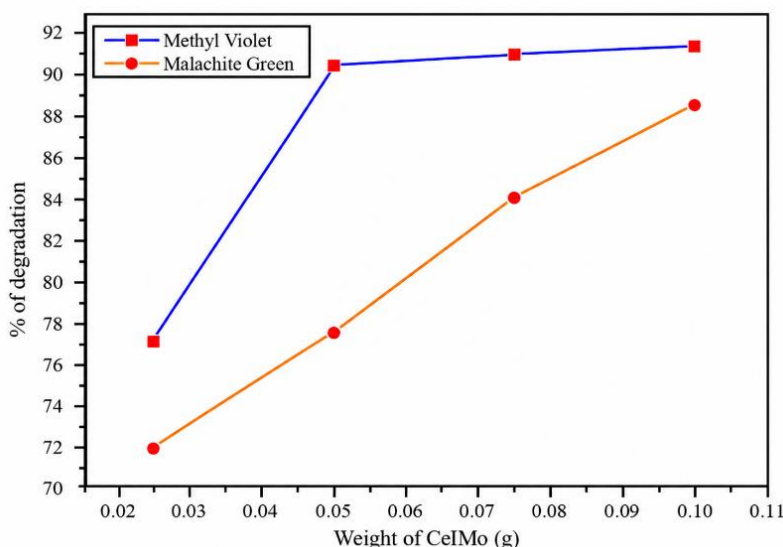


Figure 13: Plot of % dye removal vs ion exchanger dose

CeImo's efficiency in removing methyl violet and Malachite green from aqueous solution indicates its promising use in dye-containing wastewater. CeImo showed very good visible results, removing the violet and green coloration of the dye solution, and achieved about 92% dye removal after 30 min.

5. Conclusion

X-ray diffraction and scanning electron microscopy studies revealed that Ce(IV) iodomolybdate is an amorphous inorganic ion exchanger with irregularly shaped particles. Fourier transform infrared transmission spectra confirmed the presence of interstitial water, metal-hydroxyl groups, and metal-oxygen linkages in the sample. Its initial pH of 1.96 indicates that it is an effective cation exchanger. Exchange ability depends on the size and charge of the hydrated ions. K^+ is more vital than Na^+ , and Ba^{2+} is more important than Sr^{2+} . The exchange is suggested to occur at sodium chloride concentrations above 0.4 M and after 90 minutes of equilibration. The chemical is stable in distilled water. It is slightly soluble in several organic solvents and in acetic acid (2 M), but unstable in sodium hydroxide (2 M). Distribution studies show strong attraction for harmful heavy metals, as well as for the ions ultimately responsible for hardness. Specifically, the order of affinity is as follows: Pb^{2+} is the highest, followed by Ca^{2+} , Ni^{2+} , Cu^{2+} , and Mg^{2+} . It is therefore a very effective method of removing lead and calcium from filthy or hard water. The material is expected to be a good catalyst and may also help degrade toxic organic colors such as methyl violet and malachite green dyes in wastewater.

Acknowledgment: The authors gratefully acknowledge Saint Joseph University, Aditanar College of Arts and Science, Ibn Tofail University, the University of Johannesburg, and the National Research Center for their valuable academic support, guidance, research resources, and institutional assistance throughout this research.

Data Availability Statement: The data supporting this study are available from the corresponding author and may be provided upon reasonable request.

Funding Statement: No external funding or financial assistance was received for conducting this research or preparing this manuscript.

Conflicts of Interest Statement: The authors declare that there are no conflicts of interest related to this study. This manuscript represents an original contribution, and all sources of information used have been properly acknowledged through appropriate citations and references.

Ethics and Consent Statement: The study was conducted in accordance with ethical guidelines. Participants were assured that their responses would be kept confidential and anonymous.

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