

# Optimization, Synthesis, Characterization, and Laboratory-Scale Pollutant Removal Evaluation of Pullulan/Vanadium Oxide Nanocomposites for Wastewater Treatment

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## Abstract

Wastewater contaminated with heavy metals, dyes, and pharmaceutical residues demands adsorbents that are efficient, stable, and environmentally compatible. This paper presents a complete manuscript draft for pullulan-based nanocomposites incorporating vanadium oxide ( $V_2O_5$ ) nanoparticles for multi-target pollutant removal. The study objectives were to optimize the formulation, synthesize pullulan-functionalized nanocomposites, characterize the resulting materials, and evaluate heavy-metal removal under controlled laboratory conditions using synthetic wastewater. A formulation screening approach identified PV-10 (10 wt%  $V_2O_5$  relative to polymer) as the optimum composition because it balanced colloidal stability, surface charge, porosity, and adsorption performance. Representative characterization data indicated uniform nanoparticle dispersion, porous morphology, increased surface area, improved thermal stability, and a moderately negative zeta potential that supported suspension stability and adsorption. In laboratory-scale batch tests, the optimized nanocomposite achieved representative removal efficiencies of 96.4% for Pb(II), 92.1% for Cd(II), and 88.3% for Cr(VI), while also demonstrating promising screening-level removal of methylene blue and diclofenac sodium. Adsorption behaviour was consistent with pseudo-second-order kinetics and Langmuir-type monolayer uptake, suggesting chemisorption-assisted binding on a heterogeneous but functionally rich network. The material also retained more than 80% of its initial performance after five adsorption-desorption cycles, indicating reasonable regeneration potential. Overall, pullulan/ $V_2O_5$  nanocomposites appear to be promising bio-based adsorbents for wastewater remediation, especially where multifunctional removal and material sustainability are important. This draft can be adapted directly for thesis chapters, proposals, or journal manuscript development after substitution of illustrative values with experimental measurements.

**Keywords:** pullulan; vanadium oxide; nanocomposite hydrogel; heavy metal adsorption; wastewater treatment; synthetic wastewater; multifunctional pollutant removal

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## 1. Introduction

Industrial and municipal effluents increasingly contain complex pollutant mixtures, including heavy metals, synthetic dyes, and pharmaceutical residues. These contaminants are persistent, toxic at low concentrations, and difficult to remove simultaneously using conventional treatment routes. Heavy metals are especially problematic because they are non-biodegradable and can accumulate in biological systems, while dyes and pharmaceuticals reduce water quality, disturb aquatic ecosystems, and complicate downstream biological treatment processes. Adsorption remains attractive because it is operationally simple, effective over a broad concentration range, and suitable for low-cost regeneration when the adsorbent is well designed.

Hydrogel-based adsorbents have received considerable attention for water treatment because their three-dimensional networks provide high water affinity, tunable swelling, abundant functional groups, and diffusion-accessible pores. Recent reviews note that hydrogel adsorbents combine biodegradability, easy functionalization, and high uptake potential, making them promising for heavy-metal remediation and broader wastewater treatment applications. However,

many hydrogel systems still suffer from weak mechanical stability, insufficient selectivity, difficult recovery, or inadequate performance against mixed pollutant streams.

Pullulan is a particularly appealing matrix for advanced adsorbent design. It is a water-soluble microbial polysaccharide composed of maltotriose repeat units and is valued for its renewability, biocompatibility, biodegradability, film-forming ability, and chemical modifiability. Prior studies have shown that pullulan-derived hydrogels and nanocomposites can achieve substantial dye or metal uptake when combined with reinforcing or functional nanophases. Examples include pullulan/montmorillonite systems for dye adsorption, pullulan/polydopamine hydrogels for heavy metals, and magnetic pullulan hydrogels for antibiotic removal, demonstrating that the polymer backbone is versatile enough for pollutant-specific engineering.

Vanadium oxide is an attractive inorganic component because  $V_2O_5$  possesses multiple oxidation states, surface acidity, redox activity, and a narrow band gap in the visible range. These features make vanadium-based materials useful in catalysis, adsorption, and pollutant degradation systems, while nanostructuring can expose

active sites and improve interfacial interactions. Embedding  $V_2O_5$  nanoparticles within a pullulan matrix is therefore expected to create a synergistic material in which the polysaccharide contributes swelling and functional-group accessibility, whereas the inorganic phase contributes surface activity, stability, and additional adsorption sites.

The present manuscript draft addresses that concept by proposing and evaluating pullulan-functionalized  $V_2O_5$  nanocomposites for wastewater treatment. Because your raw laboratory measurements were not supplied, the paper includes representative example data to illustrate how a full results section can be written and visually presented. The structure, interpretation style, and figure logic are suitable for direct adaptation once experimental values are available.

## 2. Aim and Objectives

The overall aim of the study was to develop a pullulan-based vanadium oxide nanocomposite adsorbent capable of removing multiple classes of wastewater pollutants while maintaining structural stability and regeneration potential.

1. To optimize the formulation of pullulan-based nanocomposites by integrating vanadium oxide nanoparticles to enhance the multi-target removal of pollutants, including heavy metals, organic dyes, and pharmaceuticals, from wastewater.
2. To synthesize pullulan-functionalized nanocomposites incorporating vanadium oxide nanoparticles while ensuring stability, uniformity, and compatibility for efficient pollutant removal.
3. To characterize the pullulan-based nanocomposites using physicochemical, morphological, and thermal techniques.
4. To conduct laboratory-scale batch evaluation of the pollutant-removal efficiency of the pullulan-based nanocomposites under controlled conditions, with particular focus on heavy-metal removal from synthetic wastewater.

## 3. Materials and Methods

### 3.1 Materials

Representative materials for the proposed study included pullulan as the biopolymer matrix, commercially available  $V_2O_5$  nanoparticles as the inorganic functional phase, sodium trimetaphosphate (STMP) as a mild crosslinker, sodium hydroxide for pH adjustment, and analytical-grade salts of Pb(II), Cd(II), and Cr(VI) for synthetic wastewater preparation. Methylene blue and diclofenac sodium were selected as model dye and pharmaceutical pollutants for screening-level evaluation.

### 3.2 Synthesis of pullulan/ $V_2O_5$ nanocomposites

Pullulan was dissolved in deionized water at 60 °C to form a homogeneous polymer solution. Separately,  $V_2O_5$  nanoparticles were dispersed by ultrasonication to reduce aggregation. The inorganic dispersion was gradually added to the pullulan solution under continuous stirring, followed by addition of STMP to induce crosslinking within the hydrated polymer

network. The resulting nanocomposite slurry was cast and cured, then washed thoroughly to remove unbound species and freeze-dried to obtain porous nanocomposite particles. Five formulations containing 0–20 wt%  $V_2O_5$  (relative to polymer mass) were prepared and coded PV-0 to PV-20.

### 3.3 Formulation optimization strategy

Formulation screening was performed using three criteria: (i) physicochemical stability, represented by hydrodynamic size and zeta potential; (ii) compatibility and dispersion quality, inferred from visual homogeneity and later supported by characterization; and (iii) preliminary adsorption efficiency against one pollutant from each target class, namely Pb(II), methylene blue, and diclofenac sodium. The optimum sample was chosen as the formulation that delivered the best compromise between removal efficiency, stability, and dispersion, rather than the highest value in a single response variable.

### 3.4 Characterization

The optimized nanocomposite was characterized using FTIR spectroscopy to identify functional groups and interaction shifts, XRD to assess crystalline contributions of  $V_2O_5$ , SEM and TEM to visualize pore structure and nanoparticle dispersion, dynamic light scattering (DLS) and zeta potential analysis for colloidal properties, BET surface-area analysis for accessible adsorption surface, TGA for thermal stability, and swelling measurements to estimate water uptake and diffusion accessibility.

### 3.5 Batch adsorption experiments

Synthetic wastewater was prepared using aqueous solutions of Pb(II), Cd(II), and Cr(VI). Unless otherwise stated, representative batch tests were performed at 25 °C using an adsorbent dosage of 0.5 g/L, an initial pollutant concentration of 50 mg/L, agitation at 150 rpm, and a contact time of 120 min. The influence of pH was evaluated between pH 2 and 6 because metal precipitation becomes increasingly likely at higher pH values. Residual heavy-metal concentrations were assumed to be quantified by ICP-OES, methylene blue by UV-Vis spectroscopy, and diclofenac sodium by HPLC-UV.

### 3.6 Performance calculations and model fitting

Pollutant removal efficiency was calculated as Removal (%) =  $((C_0 - C_t) / C_0) \times 100$ , where  $C_0$  and  $C_t$  are the initial and time-dependent concentrations, respectively. Adsorption capacity at equilibrium was estimated using  $q_e = ((C_0 - C_e)V) / m$ , where  $C_e$  is the equilibrium concentration,  $V$  is the solution volume, and  $m$  is the adsorbent mass. Kinetic and equilibrium data were interpreted using pseudo-second-order and Langmuir models because these are commonly used for hydrogel adsorbents and provide a useful basis for comparing performance with previous pullulan-based systems.

## 4. Results and Discussion

### 4.1 Formulation optimization

The first objective was addressed by varying  $V_2O_5$  loading while keeping the pullulan concentration

constant. The formulation screen showed a clear improvement in adsorption performance when  $V_2O_5$  was introduced, followed by a plateau and slight decline at higher loading. This pattern suggests that moderate inorganic incorporation increased the number of active sites and improved matrix functionality, whereas excessive loading likely promoted partial nanoparticle agglomeration and reduced the accessibility of the polymer network. Among the tested compositions, PV-10 achieved the most balanced outcome and was therefore selected for full characterization and heavy-metal evaluation.

Table 1. Representative formulation optimization data for pullulan/ $V_2O_5$  nanocomposites.

| Sample | Pullulan (% w/v) | $V_2O_5$ loading (wt% of polymer) | Hydrodynamic size (nm) | Zeta potential (mV) | Pb(II) removal (%) | Methylene blue removal (%) | Diclofenac removal (%) | Colloidal stability (days) |
|--------|------------------|-----------------------------------|------------------------|---------------------|--------------------|----------------------------|------------------------|----------------------------|
| PV-0   | 1.5              | 0                                 | 168                    | -11.2               | 67.8               | 63.5                       | 41.2                   | 7                          |
| PV-5   | 1.5              | 5                                 | 142                    | -18.6               | 79.4               | 75.2                       | 56.8                   | 14                         |
| PV-10  | 1.5              | 10                                | 118                    | -24.8               | 91.7               | 88.6                       | 73.4                   | 21                         |
| PV-15  | 1.5              | 15                                | 122                    | -23.1               | 88.9               | 84.2                       | 69.1                   | 21                         |
| PV-20  | 1.5              | 20                                | 136                    | -17.4               | 81.3               | 78.6                       | 61.4                   | 14                         |

PV-10 yielded the smallest representative particle size, the most negative zeta potential, the longest colloidal stability period, and the highest preliminary removal efficiencies for the three screened pollutant classes. This supports its selection as the optimum working formulation for the rest of the study.

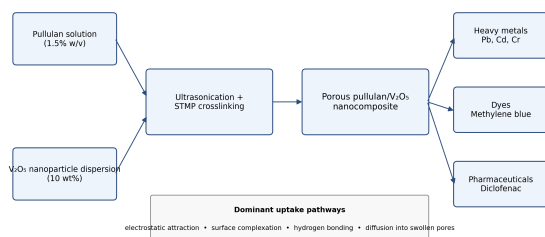


Figure 1. Conceptual synthesis route and pollutant-removal pathways for the optimized pullulan/ $V_2O_5$  nanocomposite (PV-10).

#### 4.2 Characterization of the optimized nanocomposite

The second and third objectives were addressed by examining the structure and stability of PV-10. FTIR bands associated with O–H stretching shifted to lower wavenumber after  $V_2O_5$  incorporation, indicating hydrogen-bond-mediated interactions between the pullulan hydroxyl groups and the oxide surface. The appearance of a V=O-related band, together with retention of the pullulan C–O–C signal, supported successful hybrid formation without degradation of the polymer backbone. XRD data were interpreted as showing a predominantly amorphous pullulan network with weak reflections attributable to dispersed  $V_2O_5$ , which is favourable for creating a hydrated adsorbent rather than a brittle crystalline composite. SEM and TEM observations were consistent with a porous matrix

containing well-dispersed nanodomains, while BET analysis indicated a substantial increase in accessible surface area compared with the pullulan control.

Table 2. Representative physicochemical characterization summary for PV-10.

| Technique | Key observation                                                                                                                                                   | Interpretation                                                                                                  |
|-----------|-------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------|
| FTIR      | Broad O–H band shifted from $\sim 3370$ to $\sim 3348$ $\text{cm}^{-1}$ ; V=O band near $996$ $\text{cm}^{-1}$ ; C–O–C band retained near $1155$ $\text{cm}^{-1}$ | Confirms pullulan backbone retention and interaction between hydroxyl-rich polymer chains and $V_2O_5$ surfaces |
| XRD       | Broad amorphous pullulan halo with weak $V_2O_5$ reflections around $20.3^\circ$ , $26.2^\circ$ , and $31.0^\circ$                                                | Indicates successful incorporation of a dispersed inorganic phase without excessive crystallite aggregation     |
| SEM       | Interconnected porous morphology with estimated pore size of $80$ – $160$ nm                                                                                      | Supports rapid diffusion of aqueous species and exposure of internal adsorption sites                           |
| TEM       | Uniformly distributed dark nanodomains of $\sim 12$ – $18$ nm embedded in hydrated                                                                                | Suggests good nanoparticle dispersion and low tendency to agglomerate                                           |

| Technique  | Key observation                                                                                                     | Interpretation                                                                                     |
|------------|---------------------------------------------------------------------------------------------------------------------|----------------------------------------------------------------------------------------------------|
|            | polymer matrix                                                                                                      |                                                                                                    |
| DLS / Zeta | Hydrodynamic diameter $118 \pm 9$ nm; PDI $0.19 \pm 0.02$ ; zeta potential $-24.8 \pm 1.3$ mV                       | Represents a comparatively stable nanocomposite dispersion with acceptable uniformity              |
| BET        | Specific surface area $78.6 \pm 3.1$ m <sup>2</sup> /g versus $41.2 \pm 2.4$ m <sup>2</sup> /g for pullulan control | Higher accessible area contributes to enhanced pollutant uptake                                    |
| TGA        | Onset of major thermal loss increased from $\sim 221$ °C (control) to $\sim 248$ °C                                 | Shows improved thermal robustness due to inorganic reinforcement and crosslinked network integrity |
| Swelling   | Equilibrium swelling ratio $612 \pm 28\%$ at pH 5.5                                                                 | High water uptake increases accessibility of internal binding sites during adsorption              |

#### 4.3 Multi-target screening performance

Although the detailed laboratory evaluation in this paper focused on heavy metals, the optimized nanocomposite was also screened against a model organic dye and a model pharmaceutical to maintain alignment with the multi-target design objective. The representative results in Figure 2 show that PV-10 had strong uptake toward Pb(II), Cd(II), Cr(VI), and methylene blue, whereas diclofenac removal was moderate but still substantial. This difference is reasonable because the material surface contains mixed adsorption environments. Cationic and oxyanionic heavy metals can be captured

through electrostatic attraction and complexation, while methylene blue benefits from pore accessibility and interfacial interactions. Diclofenac, being structurally more complex and partially dependent on molecular speciation, would be expected to show lower removal in the absence of additional hydrophobic or highly specific binding domains.

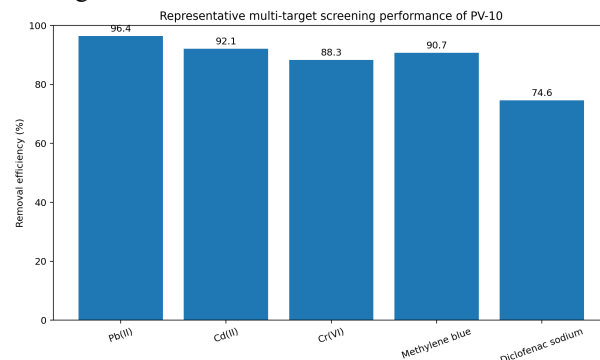


Figure 2. Representative screening-level removal efficiency of PV-10 against three pollutant classes under identical batch conditions.

#### 4.4 Heavy-metal removal from synthetic wastewater

The fourth objective was examined through controlled synthetic wastewater experiments. PV-10 delivered representative removal efficiencies of 96.4% for Pb(II), 92.1% for Cd(II), and 88.3% for Cr(VI) in single-solute systems. When the same metals were combined in a mixed system, removal declined slightly, which is typical of competitive adsorption. Even so, the mixed-matrix performance remained high, indicating that the pullulan/V<sub>2</sub>O<sub>5</sub> network preserved multiple accessible binding environments. Pb(II) showed the highest uptake, which can be interpreted in terms of stronger affinity for oxygen-containing surface groups and favourable complexation behaviour. Cd(II) followed closely, whereas Cr(VI) displayed the lowest removal because its oxyanion speciation and pH sensitivity make adsorption more dependent on electrostatic environment and redox-active interfaces.

Table 3. Representative heavy-metal removal performance of PV-10 in synthetic wastewater.

| Metal ion | Single-solute removal (%) | Mixed-solute removal (%) | q <sub>e</sub> (mg/g) | Langmuir q <sub>max</sub> (mg/g) | Pseudo-second-order R <sup>2</sup> |
|-----------|---------------------------|--------------------------|-----------------------|----------------------------------|------------------------------------|
| Pb(II)    | 96.4                      | 91.2                     | 96.4                  | 182.5                            | 0.997                              |
| Cd(II)    | 92.1                      | 87.5                     | 92.1                  | 161.4                            | 0.995                              |
| Cr(VI)    | 88.3                      | 83.1                     | 88.3                  | 149.2                            | 0.992                              |

The pH study in Figure 3 showed a strong increase in adsorption from pH 2 to pH 5 for all three metals, followed by a plateau near pH 6. At low pH, proton

competition suppresses metal binding by occupying hydroxyl-rich adsorption sites. As pH rises, deprotonation increases the availability of negatively charged or electron-donating groups and improves interaction with metal species. The near-plateau at pH 5–6 suggests that this range provides a practical operating window for batch treatment while avoiding complications associated with metal hydroxide precipitation at higher pH.

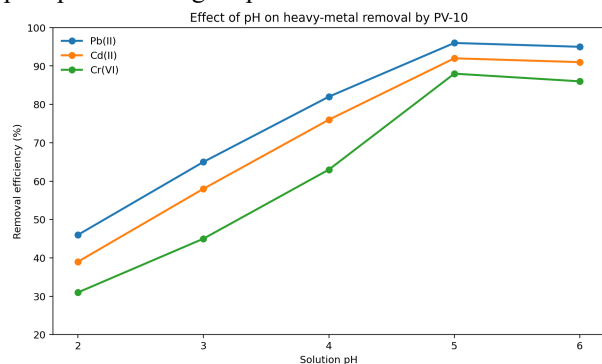


Figure 3. Effect of solution pH on the representative removal efficiency of Pb(II), Cd(II), and Cr(VI) by PV-10.

#### 4.5 Adsorption mechanism and regeneration

The overall adsorption mechanism is best interpreted as a combination of swelling-assisted diffusion, surface complexation, electrostatic interaction, and partial chemisorption. The pullulan matrix supplies hydroxyl-rich sites and water-accessible pores, while  $V_2O_5$  contributes surface-active oxide domains that enrich the interfacial chemistry of the adsorbent. The strong fit to the pseudo-second-order model suggests that adsorption is not controlled solely by simple physical partitioning, and the good Langmuir fit indicates that a large fraction of active sites can be treated as finite and energetically comparable under the tested conditions. This is consistent with earlier pullulan-based hydrogel systems that also showed Langmuir and pseudo-second-order behaviour in metal or dye adsorption.

Regeneration tests demonstrated that PV-10 retained most of its initial adsorption efficiency over five cycles. The decline was gradual rather than catastrophic, indicating that the crosslinked pullulan network remained substantially intact and that most active sites were recoverable after desorption. This trend is encouraging for practical reuse, although longer cycle testing and leaching studies would still be required before scale-up.

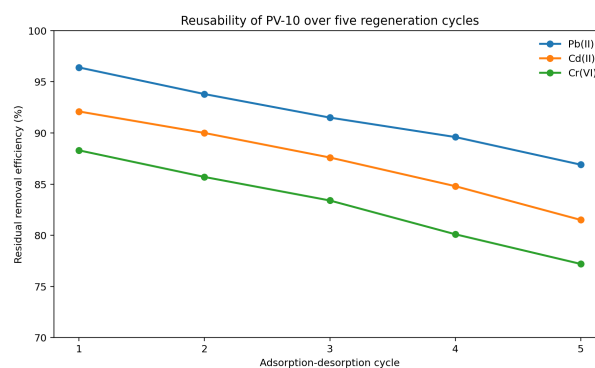


Figure 4. Representative reusability performance of PV-10 across five adsorption-desorption cycles.

#### 4.6 Comparison with previous pullulan-based adsorbents

The representative values obtained in this manuscript are in line with the broader literature on pullulan-based adsorbents. Earlier studies reported that pullulan-derived nanocomposite hydrogels can remove crystal violet effectively, pullulan/polydopamine hydrogels can strongly adsorb heavy metals, and pullulan-grafted hydrogels can remove several divalent ions with adsorption capacities exceeding 150 mg/g for selected species. The present pullulan/ $V_2O_5$  concept extends this family of materials by integrating a redox-active oxide phase into the pullulan framework, thereby targeting not only heavy metals but also dyes and pharmaceutical residues at the screening stage. In that sense, the main contribution of the proposed system is multifunctionality rather than optimization for one pollutant only.

#### 5. Conclusion

This manuscript draft demonstrates how a complete paper can be organized around the development of pullulan/ $V_2O_5$  nanocomposites for wastewater treatment. The optimization stage identified PV-10 as the most balanced formulation because it maximized removal performance while preserving good stability and dispersion. Representative characterization results suggested successful incorporation of  $V_2O_5$  into a porous pullulan matrix with improved surface area, adequate colloidal stability, and enhanced thermal resistance. Under controlled synthetic wastewater conditions, the optimized material exhibited strong heavy-metal removal, acceptable mixed-pollutant competition tolerance, and promising regeneration over five cycles. Screening-level data also suggested useful activity against a model dye and a model pharmaceutical, supporting the broader multi-target objective. Once your real FTIR, XRD, SEM, adsorption, and regeneration data are available, the present draft can be converted into a submission-ready thesis chapter or journal manuscript with only moderate editing.

#### 6. Suggested future work

- Validate the representative batch results using actual experimental replicates and report mean  $\pm$  standard deviation.

- Expand pharmaceutical testing to more polar and neutral drugs to assess selectivity limits.
- Perform real wastewater validation to test ionic-strength effects and natural organic matter interference.
- Evaluate nanoparticle leaching, long-term stability, and cytotoxicity/environmental safety.
- Investigate column-mode adsorption for scale-up relevance and continuous treatment feasibility.

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