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Synthesis of New Triazolyl Starch Derivatives for Heavy Metals Removal

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This work presents the synthesis and application of novel triazolyl starch derivatives (TABS) designed for the efficient removal of heavy metal ions from aqueous solutions. The synthetic pathway involves tosylation of natural starch, azidation, and copper-catalyzed azide–alkyne cycloaddition (CuAAC) with propargylated aromatic aldehydes to yield triazolyl-functionalized starch. Structural and morphological features were confirmed by FT-IR, SEM, and EDX analyses. The adsorption performance of TABS was evaluated under various pH values, contact times, and Pb^{2+} concentrations, showing a maximum removal efficiency of 99% and adsorption capacity of 99.0 mg g^{-1}. The enhanced affinity is attributed to chelation through triazole rings and aldehyde groups. Derived from renewable starch and demonstrating low antibacterial activity, TABS represents a promising, low-toxicity material for sustainable water purification applications.

Keywords: starch, adsorption, wastewater treatment, heavy metals removal, pollutants

Introduction

The contamination of water sources by heavy metals and organic pollutants represents a pressing global environmental issue, particularly considering increasing industrialization and the scarcity of clean water resources [1–5]. Among the strategies developed to address this challenge, adsorption has emerged as a cost-effective and efficient technique for removing a wide range of toxic substances from aqueous media. Biopolymer-based adsorbents, such as chitin, chitosan, cellulose, and starch, are especially attractive due to their natural abundance, low toxicity, and biodegradability [6–8].

Starch, a plant-derived polysaccharide composed of amylose and amylopectin chains linked by α-1,4 and α-1,6 glycosidic bonds, exhibits inherent affinity for pollutant molecules through its hydroxyl-rich structure [9,10]. However, native starch's solubility and limited number of active binding sites often restrict its direct application in water treatment. Structural modifications are commonly applied to enhance its adsorption performance, introducing functional groups capable of binding metal ions with higher affinity. This is particularly relevant for lead (Pb^{2+}), a widespread and toxic heavy metal often released into water systems from industrial activities, posing serious risks to both human and ecological health [11–13].

Recent studies have focused on modifying biopolymers by incorporating aromatic residues to improve their stability, solubility profiles, and chelation efficiency [14–16]. One promising strategy involves the attachment of aldehyde groups through linkers such as triazoles, which can serve as strong chelating moieties. Prior work has demonstrated the potential of aromatic aldehyde-based derivatives of cellulose to exhibit notable antibacterial and metal-binding properties [17–19]. However, there remains a significant gap in the systematic development of starch-based triazolyl materials specifically designed for targeted heavy metal removal.

In this study, we report the synthesis of a novel triazolyl aldehyde-functionalized starch (TABS) via click chemistry, aimed at enhancing adsorption performance toward Pb^{2+} ions. The synthetic approach utilizes 1,3-dipolar cycloaddition (Huisgen reaction) between azide-functionalized starch and a propargylated aromatic aldehyde. The resulting biopolymer is characterized and evaluated for its structure, morphology, and adsorption behavior. This research addresses the need for efficient, biodegradable adsorbents with tunable functionalities for sustainable water purification applications.

Experimental part

Materials

All chemicals used in this study were of analytical grade (typically ≥98% purity) and used without further purification. Starch powder, pyridine (≥99%), tosyl chloride (≥98%), acetone (≥99.5%), dimethylformamide (DMF, ≥99.8%), sodium azide (≥99.5%), 4-hydroxybenzaldehyde (≥98%), sodium carbonate (≥99.9%), propargyl chloride (≥97%), ethanol (≥99.5%), sodium ascorbate (≥98%), copper(I) chloride (CuCl, ≥99%), and lead(II) nitrate ($Pb(NO_3)_2$, ≥99%) were purchased from Sigma-Aldrich. Deionized water (resistivity ≥18.2 MΩ·cm) was used throughout the study for solution preparation and washing steps. Aqueous solutions of Pb^{2+} were freshly prepared by dissolving $Pb(NO_3)_2$ in deionized water to obtain a standard concentration of 50 mg L^{-1}, used in all adsorption experiments unless otherwise specified.

Instrumentation

Fourier-transform infrared (FT-IR) spectra were recorded using a Bruker Alpha II spectrometer in the range of 4000–400 cm^{-1} with samples prepared as KBr pellets. Scanning electron microscopy (SEM) images were obtained using a JEOL JSM-6610LV microscope operated at an accelerating voltage of 20 kV to analyze the surface morphology of the synthesized materials. Elemental composition was assessed by energy-dispersive X-ray spectroscopy (EDX) attached to the SEM unit. Lead ion concentrations in aqueous solutions were measured using an atomic absorption spectrophotometer (AAS), model AA-7000 (Shimadzu), equipped with a graphite furnace atomizer.

Synthesis of triazolyl aldehyde-based starch

Synthesis of tosylated starch (compound 2)

Starch (5 g, ~32 mmol of glucose units) was dissolved in 50 mL of pyridine, followed by the slow addition of tosyl chloride (7 g, ~21 mmol). The reaction mixture was stirred at room temperature for 72 h. The precipitated product was collected by filtration, washed thoroughly with acetone and water, and dried under vacuum to obtain the tosylated starch derivative (4.7 g).

Synthesis of azido-starch (compound 3)

The tosylated starch (4.7 g) was dissolved in 50 mL of DMF, and sodium azide (5 g) was added. The mixture was refluxed at 80 °C for 72 h. After cooling, the product was filtered, washed with acetone and water, and dried under vacuum to yield the azido-starch. The synthetic sequence for the tosylation and subsequent azidation of starch is illustrated in Scheme 1.

Synthesis of 4-(Prop-2-yn-1-yloxy)benzaldehyde (compound 4)

4-Hydroxybenzaldehyde (5 g), sodium carbonate (5 g), and propargyl chloride (5 g) were refluxed in 50 mL of dry acetone for 24 h under stirring. The reaction progress was monitored by thin-layer chromatography (TLC). After completion, the product was filtered, washed with cold water, and dried. The reaction is depicted in Scheme 2.

Click cycloaddition to form TABS (compound 5)

A solution of 4-(prop-2-yn-1-yloxy)benzaldehyde (0.1 g) in 40 mL of dry ethanol was mixed with azido-starch (0.2 g), sodium ascorbate (0.1 g), and CuCl (0.1 g). The mixture was stirred at room temperature for 72 h under a nitrogen atmosphere. The precipitate was filtered, extensively washed with acetone and water, and dried under vacuum to obtain the final triazolyl aldehyde-functionalized starch (TABS).

Adsorption experiments

General procedure

Adsorption performance toward Pb^{2+} ions was evaluated using a batch depletion method. The amount of adsorbed ions (Q, mg g^{-1}) was calculated using the equation:

$Q = (C_0 - C_t) \times V/m$,

where C_0 and C_t are the starting and remaining concentration of Pb^{2+} ions in the solution (mg L^{-1}); V is the volume of the solution (L); m is the mass of the grafting starch (g), respectively.

Effect of initial Pb^{2+} concentration

Different masses of TABS (0.02–0.1 g) were added to 10 mL of Pb^{2+} solution. The mixtures were gently shaken for 8 h at room temperature. Pb^{2+} concentration in the supernatant was measured using atomic absorption spectroscopy (AAS). Optimal performance was observed with 0.06 g of adsorbent.

Effect of contact time

TABS (0.06 g) was added to 10 mL of 50 mg L^{-1} Pb^{2+} solution. Samples were collected at pre-determined time intervals and analyzed to determine the adsorption kinetics.

Effect of pH

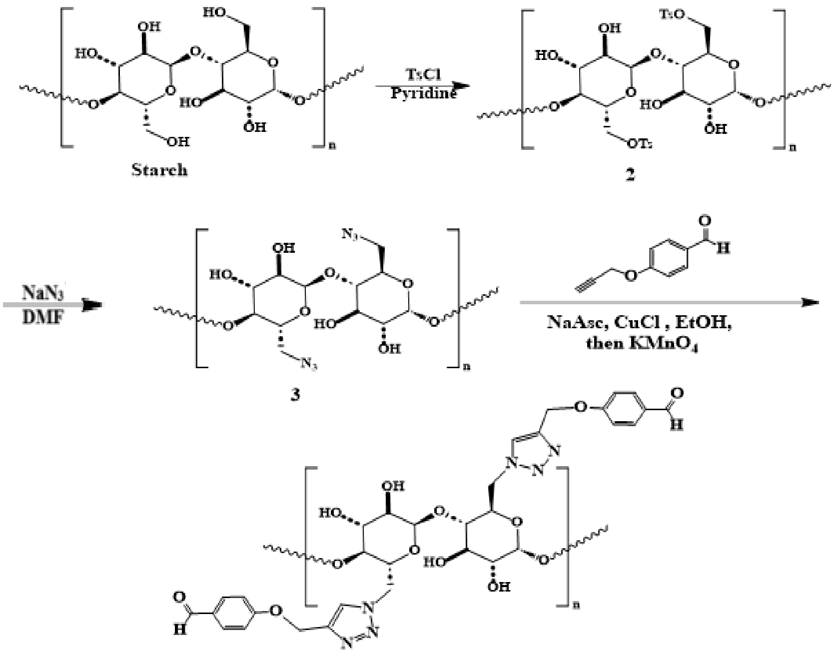
The pH of the Pb^{2+} solution was adjusted from 2 to 7 using 0.1 M HCl or NaOH. Adsorption experiments were performed under the same conditions. For complexation control, hydroxylamine hydrochloride (1.5 mL) was added at pH 3.2, and excess reagent was back titrated. All experiments were conducted in triplicate.

Results and Discussion

Synthesis

The synthetic route toward the triazolyl aldehyde-based starch (TABS) is outlined in Scheme 1, which involves three main steps: tosylation of native starch, conversion to azide, and subsequent CuAAC click cycloaddition with an alkyne-functionalized aromatic aldehyde. The tosylation selectively activates the primary hydroxyl groups of the glucose units, facilitating regioselective substitution. The resulting tosylated starch (compound 2) is then treated with sodium azide in DMF to afford the azide-functionalized starch (compound 3). Separately, 4-hydroxybenzaldehyde is converted into 4-(prop-2-yn-1-yloxy)benzaldehyde (compound 4) with propargyl chloride in the presence of potassium carbonate under reflux in acetone.

The final Cu(I)-catalyzed azide–alkyne cycloaddition (CuAAC) between compounds 3 and 4 yields the desired triazolyl aldehyde-functionalized starch derivative (compound 5, TABS).



Scheme 1. Synthesis of triazolyl aldehyde-based starch (TABS) polymer.

Table 1. General properties of triazolyl aldehyde-based starch (TABS).

Polymer	Color	Appearance	Potential Applications	Toxicity	Solubility
TABS	Pale-Yellow	Solid	Chiral separation, drug delivery, solid–liquid extraction	*E. coli:* 8 (0.01 mM), 10 (0.05 mM), 6 (0.1 mM) *S. aureus:* 6 (0.01 mM), 8 (0.05 mM), 4 (0.1 mM)	DMSO, DMF (+++); EtOH, MeOH (+--); DCM, toluene, hexane (---)

Note: (+++) = fully soluble, (+--) = partially soluble, (---) = insoluble.

Characterization of TABS

The obtained TABS polymer was a pale-yellow solid, amorphous in appearance. Table 1 summarizes its solubility behavior and antibacterial response. As expected, due to its highly polar backbone rich in hydroxyl and triazole functionalities, the polymer exhibited complete insolubility in non-polar solvents such as toluene, hexane, and dichloromethane. Conversely, full solubility was observed in polar aprotic solvents (DMF, DMSO), and partial solubility was noted in polar protic solvents such as methanol and ethanol.

Biological activity surveys revealed weak antibacterial effects, with *E. coli* and *S. aureus* showing inhibition zones <10 mm at concentrations ≥0.01 mM, indicating low cytotoxicity and potential biocompatibility.

Fourier-transform infrared (FT-IR) spectroscopy (Figure S1) was used to monitor the chemical transformations at each stage. The azide-functionalized starch exhibited a strong stretching band at 2034 cm^{-1}, characteristic of the $–N_3$ group. After the click reaction, this band disappeared, confirming triazole formation. New bands at 1628 and 1643 cm^{-1} were attributed to C=N and C=C stretching vibrations in the triazole ring, confirming successful cycloaddition.

The surface morphology of the TABS material was investigated by SEM (Figure S2). The images revealed irregular and porous structures with sponge-like morphology and aggregated domains, potentially favoring adsorption processes. EDX analysis (Figure S3) confirmed the presence of C, O, and N atoms, consistent with the modified starch framework and the formation of the triazole ring.

Optimization of adsorption parameters

Effect of adsorbent dosage

To investigate the effect of TABS dosage on Pb^{2+} removal, varying masses of polymer (0.02, 0.04, 0.06, 0.08, and 0.1 g) were added to 10 mL of a 50 mg L^{-1} $Pb(NO_3)_2$ solution and stirred gently at room temperature for 8 hours. After equilibrium, the Pb^{2+} concentration in the supernatant was determined via atomic absorption spectrophotometry (AAS). As shown in Figure 4, an increase in adsorbent mass led to greater Pb^{2+} removal efficiency, with optimal adsorption (~99.9%) observed at 0.06 g. Beyond this point, the effect plateaued due to saturation of available binding sites.

Effect of contact time

The adsorption kinetics were examined by monitoring Pb^{2+} uptake over time. TABS (0.06 g) was added to 10 mL of 50 mg L^{-1} Pb^{2+} solution and stirred at room temperature. Aliquots were collected at different time intervals. As illustrated in Figure S5, the adsorption process was rapid within the first 8 hours, reaching near-equilibrium thereafter, indicating strong affinity and efficient metal-ion binding by the TABS structure.

Effect of pH

The influence of pH on Pb^{2+} adsorption was assessed in the range of pH 2–7. The solution pH was adjusted using 0.1 M HCl or NaOH prior to the addition of 0.06 g of TABS. For accuracy, hydroxylamine hydrochloride (1.5 mL) was used as a complexing agent at pH 3.2, with excess reagent titrated using 0.1 M HCl. Each experiment was performed in triplicate. As presented in Figure 6, adsorption efficiency increased sharply with rising pH, peaking around pH 5–6. At low pH (2–3), H^+ ions compete with Pb^{2+} for binding sites, reducing efficiency. At higher pH, deprotonation of active sites enhances complexation with Pb^{2+} ions.

The experimental data suggests characteristics consistent with monolayer chemisorption. The rapid uptake of Pb^{2+} within the first 8 hours and the saturation trend observed at higher adsorbent dosages are indicative of a finite number of active sites with strong affinity for metal ions, as typically described by the Langmuir isotherm model. Moreover, the fast initial adsorption followed by equilibrium plateau supports the involvement of chemically specific interactions, such as coordination between Pb^{2+} ions and nitrogen atoms in the triazole rings. This behavior aligns qualitatively with pseudo-second-order kinetic models, which are commonly associated with chemisorption mechanisms involving valence forces through electron sharing.

Adsorption performance and comparison with literature

Under optimized conditions (0.06 g adsorbent, pH ~5, 8 h contact time), TABS demonstrated over 99% removal of Pb^{2+}, corresponding to an adsorption capacity of 99.0 mg g^{-1}. This high efficiency is attributed to the presence of nitrogen-rich triazole rings capable of coordinating Pb^{2+} ions, further stabilized by aromatic aldehyde moieties on the starch backbone. The proposed mechanism involves coordination of Pb^{2+} to the lone pair electrons on the nitrogen atoms of the triazole moieties, forming stable chelate complexes. Additionally, adjacent aldehyde groups may contribute to metal binding through electrostatic interactions or secondary coordination, enhancing the overall sorption capacity of the biopolymer.

Table 2 provides a comparative overview of TABS performance relative to other starch-based adsorbents. TABS exhibited superior or comparable efficiency, especially when considering that many alternatives involve more complex synthetic procedures or achieve lower removal percentages.

Conclusion

In this study, a novel triazolyl aldehyde-functionalized starch (TABS) was successfully synthesized via a multi-step approach involving tosylation, azidation, and Cu(I)-catalyzed azide–alkyne cycloaddition. Structural and morphological characterizations confirmed the successful incorporation of triazole and aldehyde moieties into the starch backbone. The material exhibited favorable solubility in polar aprotic solvents and demonstrated low antibacterial activity, indicating potential for safe environmental application.

Adsorption experiments revealed that TABS effectively removed Pb^{2+} ions from aqueous solutions, achieving a maximum removal efficiency of 99% and an estimated adsorption capacity of 99.0 mg g^{-1} under optimized conditions. The adsorption performance was influenced by solution pH, contact time, and adsorbent dosage, with fast kinetics and saturation behavior indicative of chemisorption. Qualitative analysis suggested that the adsorption mechanism likely involves coordination between Pb^{2+} ions and nitrogen atoms of the triazole rings.

Table 2. Comparison of TABS with other starch-based adsorbents for Pb^{2+} removal.

Absorbent	Adsorption capacity mg g^{-1}	Adsorption percentage, %	Reference
Crosslinked amphoteric starch (CAS) 1	21.01	78	[20]
Crosslinked amphoteric starch (CAS) 2	62.11	NR	[21]
DASU (Dialdehyde starch urea)	37.9	35	[22]
DASTU (Dialdehyde starch thiourea)	19.6	18	[22]
DASAPyZO (Dialdehyde starch aminopyrazole)	37.0	74	[23]
Starch-g-acrylic acidcopolymer	35.2	50	{24]
Dithiocarbonate modified starch	1.3	75	[25]
Carboxymethyl starch–acid hydrogel	110.0	93	[26]
Crosslinked amino starch	1.2	NR	[27]
TABS (current work)	99.0	99	current work

Compared to other starch-based adsorbents reported in the literature, TABS displayed competitive or superior performance, while benefiting from a straightforward synthetic route and renewable raw materials. These findings support the potential of TABS as a promising biodegradable and efficient adsorbent for heavy metal remediation in water treatment applications.

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