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Polyethyleneimine-derived dithiocarbamates for Pb(II) stabilization in MSWI fly ash leachate: Mechanisms, stability and economic assessment☆

Jinfeng Tang [a,h,1], Yanyan Li [a,1], Xinmei Lin [a], Minhua Su [a,*], Lizhi Tong [b,*], Yun Zhu [c], Biming Chen [c], Benhua Liao [c], Małgorzata Szlachta [d], Kaimin Shih [e], Junhua Xu [f,g,*]

[a] Linköping University - Guangzhou University Research Center on Urban Sustainable Development, School of Environmental Science and Engineering, Guangzhou University, Guangzhou 510006, China
[b] National Key Laboratory of Water Environmental Simulation and Pollution Control, South China Institute of Environmental Sciences, Ministry of Ecology and Environment (MEE), Guangzhou 510655, China
[c] Guangzhou City Management Technology Research Center, 510055, Guangzhou, China
[d] Faculty of Engineering and Natural Sciences, Tampere University, P.O. Box 541, FI-33104 Tampere, Finland
[e] Department of Civil Engineering, The University of Hong Kong, SAR, China
[f] Geological Survey of Finland, P.O. Box 96, FI-02151 Espoo, Finland
[g] PureTech Industry Oy, P.O. Box 31, FI-70600 Kuopio, Finland
[h] Nuclear Chemistry and Industrial Material Recycling, Department of Chemistry and Chemical Engineering, Chalmers University of Technology, 412 96, Gothenburg, Sweden

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ABSTRACT

Municipal solid waste incineration (MSWI) fly ash is hazardous due to its high heavy metal content, particularly Pb, which is highly mobile and poses significant health risks. This study synthesized polyethyleneimine-derived dithiocarbamates (PDTCs) with multiple sulfur-containing functional groups for chelation-based Pb(II) stabilization in MSWI fly ash leachate-related systems. The dissolved Pb(II) removal efficiency exceeded 95% under optimized conditions, indicating effective chelation-based stabilization of mobile Pb(II). X-ray photoelectron spectroscopy (XPS), scanning electron microscopy (SEM), and thermogravimetric analysis (TGA) were employed to characterize the properties of Pb–PDTC precipitates and elucidate the chelation mechanism. The results demonstrate that coordination between -CSS- groups in PDTCs and Pb(II) was the main mechanism for Pb(II) chelation and stabilization. Among the tested PDTCs, PDTC-600 exhibited the best overall Pb(II) stabilization performance due to better accessibility of active -CSS- groups. Under acidic conditions, a lower pH destabilized the chelates. Despite this, PDTCs maintained high dissolved Pb(II) removal efficiency in aqueous batch systems, indicating their potential for chelation-based Pb(II) stabilization in leachate-related systems. This study provides a mild and potentially cost-effective strategy for chelation-based Pb(II) stabilization in MSWI fly ash leachate-related systems. Future research should further verify the performance of PDTCs in real, highly alkaline fly ash leachate and optimize chelator molecular weight to balance Pb(II) stabilization efficiency, precipitate stability, reagent safety, and environmental compatibility.

1. Introduction

The management of municipal solid waste incineration (MSWI) fly ash constitutes a formidable challenge, exacerbated by the rapid advancements in incineration technologies [1–3]. Owing to its elevated leaching concentrations of hazardous heavy metals such as Pb, Cr, Zn, Cu, Ni, and Cd, MSWI fly ash has been deemed hazardous waste across numerous countries and regions, including China [4,5]. Conservative estimates suggest that MSWI fly ash accounts for approximately 3–5% of the incinerated waste mass [6,7]. With the rapid expansion of China's MSW incineration capacity in recent years, the annual generation of MSWI fly ash is estimated to reach several million tons, likely in the

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* Corresponding author at: Geological Survey of Finland, P.O. Box 96, FI-02151 Espoo, Finland.
E-mail addresses: mhsu@gzhu.edu.cn (M. Su), tonglizhi@scies.org (L. Tong), junhua.xu@gtk.fi (J. Xu).

[1] These authors contributed equally to this work.

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range of 8–10 million tons, highlighting the urgent need for reliable stabilization strategies [8–10]. This considerable quantity significantly endangers the ecological environment and public health [11].

Among these toxic metals, lead (Pb) is extensively documented due to its toxicity in water, soil, and solid wastes [12,13]. Its accumulation in biological organisms and subsequent amplification through the food chain can lead to grave health issues in the general population [14]. To mitigate the presence of Pb, various remediation technologies, including adsorption [15] [16], ion exchange [17], chemical precipitation [18], and membrane separation [19], have been explored. Dithiocarbamates (DTCs), recognized for their high metal-binding affinity and cost-effectiveness, have emerged as promising chemical chelators for treating Pb-contaminated wastewater and hazardous solid wastes [20,21].

DTCs, characterized by one or more -N(H)-CSS- functional groups [22], are synthesized by converting primary or secondary amine groups using carbon disulfide in basic or alcoholic solutions [23]. Recent studies have reported an enhanced metal removal efficiency by increasing the functional groups of DTCs or modifying composites with DTCs [24,25]. Due to their abundant sulfur-containing functional groups and strong affinity toward soft heavy metals, polymeric dithiocarbamate chelating agents have attracted increasing attention for heavy metal stabilization in fly ash and related leachate systems [26–29].

Polyethyleneimine (PEI), a versatile precursor for synthesizing macromolecular DTCs [30], contains abundant reactive amine groups. Its molecular weight can vary considerably [31–33], thereby affecting the accessibility of functional groups and the chelation performance of the resulting PDTCs [34]. However, changes in molecular structure can influence performance and stability. For example, longer alkyl groups enhance metal chelation [35], while increased polymerization can reduce efficiency and affect water solubility [22]. Additionally, longer alkyl chains reduce the decomposition rates of metal chelates [36]. Despite their effectiveness, traditional synthesis methods for polymer chelators are energy-intensive and produce excessive by-products [37]. The impact of molecular weight variations or raw material types on chelation effectiveness remains underexplored.

In this study, the research scenario is defined as the chelation-based stabilization of dissolved Pb(II) in MSWI fly ash leachate-related aqueous systems, rather than the direct extraction or removal of Pb from raw fly ash solids. Simulated Pb-containing leachate was used as a controlled system to clarify the effects of polymer molecular weight, -CSS- functional groups, and coexisting ions on Pb(II) stabilization. Based on this scenario, this study aimed to: (1) synthesize a series of macromolecular DTCs with varied molecular weights; (2) evaluate the efficiency of these PDTCs in stabilizing dissolved Pb(II) in the presence of common and competing heavy metals; and (3) elucidate the reaction mechanisms and assess the environmental stability of PDTC-metal chelates [38]. To achieve this, synthesis conditions were optimized to produce two novel, soluble polymer chelating agents, emphasizing the critical role of molecular weight and amine functionality in optimizing chelation efficiency. The investigation explored the ability of these agents to stabilize dissolved Pb(II) in fly ash leachate-related aqueous systems, with a focus on chelation mechanisms, acid–base interactions, and the stability of the metal complexes formed [21,39,40]. A preliminary economic analysis was also conducted to assess the cost-effectiveness of these novel agents in heavy metal remediation. This study provides mechanistic insight into the molecular-weight-dependent performance of PDTCs for Pb(II) stabilization in MSWI fly ash leachate-related systems.

2. Experimental

2.1. Materials and ash samples

In this study, analytical grade chemicals were utilized for all experimental procedures to guarantee high precision and accuracy. Aqueous solutions were prepared using deionized water, ensuring a purity exceeding 18 MΩ/cm. For the experiments, branched polyethyleneimines (PEIs) with different molecular weights (MW = 600, 1800, and 10,000) were sourced from Shanghai Aladdin Biochemical Technology Co. Additionally, essential reagents including carbon disulfide (CS_2), ethanol (C_2H_5OH), sodium hydroxide (NaOH), and a range of metal chloride salts ($CuCl_2$, $PbCl_2$, $ZnCl_2$, $NiCl_2$, $CdCl_2$, and $CrCl_2$) were procured from reputable local suppliers. The focus of this study was to delineate the input mass concentration, which was determined based on the maximum concentration of the selected metals present in the effluents investigated. All characterization methods used in the experiments are described in the supplementary materials (SI).

Fly ash samples for this study were collected from a municipal waste incineration plant situated in Shenzhen, China. This plant employs the prevalent grate combustion technology, with a daily processing capacity of 3000 tons of waste, the plant served as an ideal source for our samples. Over a period of 6 months, fly ash samples were systematically gathered, ensuring that those used in our experiments originated from the same homogenized batch from the plant.

As outlined in Table 1, the concentrations of target elements in the ash samples were quantified using inductively coupled plasma optical emission spectroscopy (ICP-OES) and inductively coupled plasma mass spectroscopy (ICP-MS). The metal concentrations listed in Table 1 represent total metal contents after total digestion rather than leaching concentrations. After digestion, the solutions were analyzed by ICP-MS, with all measurements performed in triplicate.

2.2. Synthesis of chelating agents

Previous experimentation established the mutual solubility of PEI and carbon disulfide in ethanol across all proportions. Consequently, ethanol was selected as the solvent for the chelation reaction medium. Unlike traditional synthesis methodologies, sodium hydroxide was excluded due to its slow reaction with ethanol, which forms sodium ethoxide, a compound known for its toxicity.

The experimental protocol was as follows: A specific concentration of PEI in ethanol was prepared, stirred, and refrigerated. A measured volume was then transferred to a three-necked flask, and carbon disulfide was added incrementally with continuous stirring at ambient temperature. After complete addition, the mixture was stirred until the reaction finished. The resulting white precipitate was separated via centrifugation, washed with deionized water, and dried in a vacuum oven at 45 °C to a constant weight. This process yielded light yellow PEI chelating agents (PDTC-600, PDTC-1800, PDTC-10000, corresponding to the molecular weights of the PEI precursors). The precipitate here refers to the isolated product formed in ethanol during synthesis. Before the Pb(II) stabilization experiments, the obtained PDTC solids were prepared as stock solutions for controlled dosing.

Furthermore, this study evaluated the influence of various parameters on the yield (defined as the percentage of the actual synthesized chelating agent relative to the theoretical yield) and sulfur content in PDTC. These parameters included the reaction time (0.5, 2, 4, 6, 8, and 12 h), the molar ratio of carbon disulfide to PEI monomer (ranging from 1:0.5 to 1:1), PEI concentrations (25, 75, 125, 175, and 225 g/L), and

Table 1
Total metal concentrations in fly ash samples after total digestion (mg/kg, dry weight).

Element	Concentration (mg/kg)
Cd	196 ± 4
Cr	164 ± 4
Cu	1710 ± 62
Mn	183 ± 7
Ni	44 ± 1
Pb	1540 ± 53
Zn	9610 ± 280

reaction temperatures (10, 25, 45, and 65 °C).

2.3. Chelation-based stabilization of Pb(II) in MSWI fly ash leachate systems

To simulate dissolved Pb(II) in MSWI fly ash leachate and to isolate the effects of chelator molecular weight and coexisting ions, batch stabilization experiments were conducted using prepared Pb(II)-containing solutions under controlled pH conditions. The efficacy of the synthesized chelating agents, polyethyleneimine-derived dithiocarbamates (PDTCs), was evaluated by measuring the decrease in dissolved Pb(II) concentration and the stability of the resulting Pb–PDTC precipitates. A focal point of this analysis was the impact of the molar ratio of -CSS- groups to Pb present in PDTCs on the removal efficiency of Pb^{2+} ions. Furthermore, the influence of common coexisting ions (Na^+, K^+, Ca^{2+}, and Mg^{2+}) and trace toxic elements (Cd^{2+}, Cu^{2+}, Zn^{2+}, and Ni^{2+}) on this removal process was examined, specifically under a controlled molar ratio of -CSS- to Pb set at 2.0. The concentrations of coexisting metal ions were selected to provide controlled concentration gradients for comparing competitive effects.

In the batch precipitation experiments, a 100 mL Pb^{2+} solution (pH = 5.0) was prepared using either 0.1 mol/L HNO_3 or 0.1 mol/L NaOH. HNO_3 was used only for pH adjustment because nitrate has weak complexation with Pb(II), minimizing interference with PDTC–Pb chelation. PDTC stock solutions with a mass concentration of 10 wt% were prepared and added to the Pb(II)-containing solutions according to the designed -CSS-/Pb(II) molar ratios, which were calculated based on the sulfur contents obtained from elemental analysis. The mixture was then subjected to continuous stirring at 200 r/min for 3 min, followed by a reduced speed of 50 r/min for an additional 10 min. To avoid the potential precipitation of heavy metal hydroxides under alkaline conditions, the final pH of the solution was adjusted to 5.0 [41]. Post-stirring, the supernatant of the solution was filtered through a 0.22 μm membrane filter and subsequently diluted with 2% HNO_3. The residual metal concentrations in the solution were quantitatively determined using ICP-MS. All batch experiments were performed in triplicate.

$$\text{Removal rate}\ (\%) = (C0 - Ct)/C0 \times 100\% \quad (1)$$

where C_o and C_t are the total metal concentrations at reaction times 0 and t min, respectively.

2.4. Evaluation of the stability of chelated precipitates

The acid stability of the chelated precipitates, specifically PDTC-Pb chelates synthesized from various PDTCs, was evaluated using a method established in prior research. In this process, 0.1 g of the PDTC-Pb chelates was uniformly dispersed in 100 mL of deionized water, maintaining a solution-to-solid ratio of 1000:1. The pH of this suspension was adjusted to target values of 4.0, 5.0, 5.6, 7.0, 9.0, and 11.0, utilizing either a NaOH solution or an acidic mixture of H_2SO_4 and HNO_3 at a 2:1 mass ratio.

The concentration of Pb in the solution post-leaching was measured at time intervals of 1, 5, 15, 30, and 70 days, employing ICP-MS. This analysis facilitated the generation of a detailed time-based profile of metal leaching concentrations, effectively correlating the extent of Pb release with the pH levels of the medium.

3. Results and discussion

3.1. Synthesis and optimization of polyethyleneimine-derived dithiocarbamates (PDTCs)

The viscosity dynamics of PEI in aqueous solutions were analyzed, revealing an increase in viscosity with a higher molecular weight. Higher viscosities indicated incomplete polymer chain extension. To ensure representative optimization results, PEI with a molecular weight of 10,000 (PEI-10000) was selected for optimization of the synthesis conditions. As illustrated in Fig. 1a, the sulfur content in the synthesized chelating agent gradually increased over the reaction time but was not significantly pronounced. The yield of the chelating agent stabilized after 2 h of reaction. These results suggest that in a homogeneous reaction system with anhydrous ethanol as the medium, carbon disulfide can rapidly esterify with the amine groups on polyethyleneimine molecules. Contrary to existing literature, in which the reaction color has been reported to transition from light yellow to orange, our setup rapidly formed a white precipitate upon adding carbon disulfide, indicating a faster esterification process.

Theoretically, the ideal molar ratio between polyethyleneimine (PEI) monomers and carbon disulfide (CS_2) is 1:1. However, due to the low boiling point (45 °C) and volatility of CS_2, excess CS_2 is often used in practice. As displayed in Fig. 1b, the yield of the chelating agent and the sulfur content varied minimally with changes in the CS_2 molar ratio, indicating a rapid and efficient interaction between PEI and CS_2 in a homogeneous system. Fluctuations in yield may be due to product loss during the washing stage.

A fixed CS_2 molar ratio theoretically suggests that decreasing the PEI concentration and increasing the reaction volume should extend PEI molecular chains, enhancing the reaction efficiency. However, as illustrated in Fig. 1c, neither the chelator yield nor the sulfur content changed significantly with an increase in the PEI concentration. This indicates that for PEI with a molecular weight of 10,000, the reaction is effective across a concentration range of 25 to 225 g/L.

Regarding temperature sensitivity (Fig. 1d), the sulfur content in the chelating agent remained relatively stable between 10 °C and 45 °C, fluctuating between 32.5% and 32.9%. However, at 65 °C, the sulfur content significantly decreased to 29.6%, probably due to the exothermic nature of the PEI–CS_2 reaction. While higher temperatures typically accelerate reactions, excessively high temperatures can reverse the reaction direction, reducing the reactivity of amine groups in PEI.

These results indicate that, unlike reaction temperature, factors such as the concentration of polyethyleneimine, the reaction time, and the molar ratio exert a minimal influence on the sulfur content of the synthesized chelating agent. Notably, the yield of the chelating agent exhibited considerable variation, which is probably attributable to the potential loss of some of the product during the cleaning process. Consequently, these yield measurements should primarily be regarded as indicative.

Based on these findings, the optimal synthesis conditions for chelating agents derived from polyethyleneimines of various molecular weights were established: a titration temperature range of 5 to 10 °C, a fixed reaction temperature of 25 °C, a reaction duration of 4 h, a carbon disulfide to polyethyleneimine monomer molar ratio of 1:0.6, and a polyethyleneimine ethanol solution concentration of 225 g/L.

3.2. Characterization of PDTCs

The sulfur content is an important index to evaluate the properties of chelating agents. As can be seen from the element analysis results in Fig.2a, PEIs with different molecular weights showed similar C, H, and N contents, indicating that they have the same monomer. The sulfur content in the PDTCs was approximately 30%, corresponding to about 60% of the theoretical value calculated under the assumption of complete PEI functionalization. This result indicates incomplete functionalization under the tested conditions, but it should not be interpreted as the exact conversion ratio of primary amines because the distribution of primary, secondary, and tertiary amines was not directly quantified.

Based on the UV–Vis absorption spectra of the PDTCs (Fig. 2), maximum absorption occurred in the vicinity of 234 nm and 288 nm, which can be assigned to the π-π* transition of N…C…S groups and the n–π* transition of non-bonding electrons on sulfur atoms to the conjugated system in S…C…S groups. The analytical data indicate that the

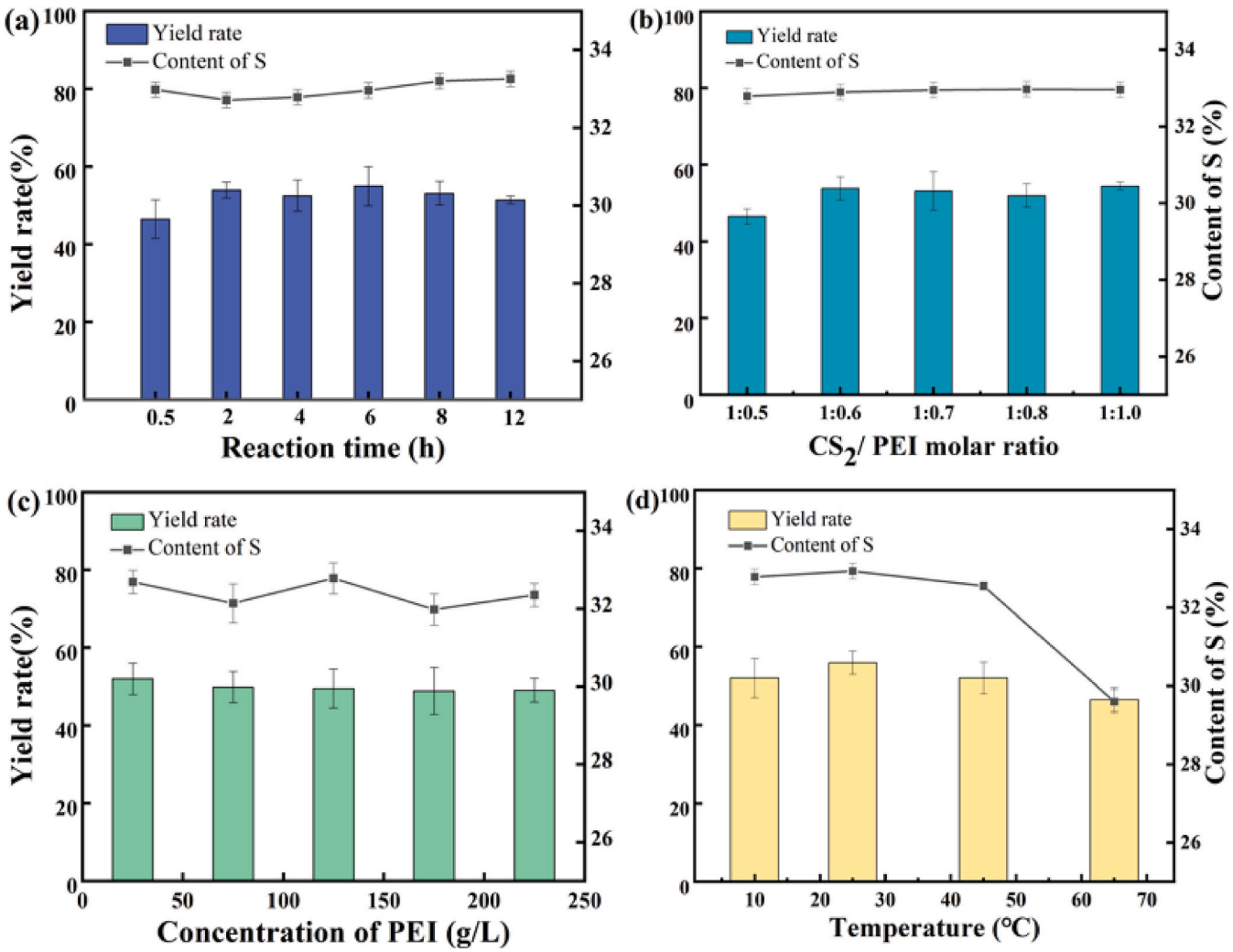

Fig. 1. Synthesis and optimization of polyethyleneimine-derived dithiocarbamates (PDTCs): effects of (a) reaction time, (b) CS_2/PEI molar ratio, (c) PEI concentration, and (d) reaction temperature.

major functional groups in PDTCs were C—N, C═S, and C—S [42].

The structures of the three PEIs and PDTCs were determined using a Thermo Nicolet 380 infrared spectrometer. As illustrated in Fig. 2**c** and **d**, the PEIs and PDTCs had similar infrared spectral characteristics and functional groups both before and after the reaction. However, the S—H weak characteristic absorption peak of PDTC-600-Pb disappeared at 2711 cm^{-1}. The area of the S—H stretching vibration absorption peak was reduced at 1624 cm^{-1}. Other than this, the positions of other absorption peaks of PDTC-600 changed little after the reaction, suggesting that the only functional group change in PDTC-600 during the reaction was -SH at 1624 cm^{-1}. The strong N—H stretching vibration absorption peak was at 3365 cm^{-1}, the C—H stretching vibration absorption peak was at 2927 cm^{-1}, the S—H weak absorption peak was at 2711 cm^{-1}, the S—H stretching vibration absorption peak was at 1624 cm^{-1}, and the C═S stretching vibration absorption peaks were at 2104 cm^{-1} and 1045 cm^{-1}, respectively. The PDTCs exhibited spectral features corresponding to a bidentate mode of binding, having a stretching vibration at 958 cm^{-1} and 1452 cm^{-1} for C—S and C—N [43], proving that there are dithiocarbamate groups in the product.

Similarly, the S—H weak characteristic absorption peaks of PDTC-1800-Pb and PDTC-10000-Pb disappeared at approximately 2700 cm^{-1} and the area of their S—H stretching vibration absorption peaks decreased at approximately 1600 cm^{-1}. Except for -SH, the functional groups of PDTC-1800-Pb and PDTC-10000-Pb changed little during the reaction. From these results, it can be inferred that the key functional group for Pb^{2+} capture by dithiocarbamate-type compounds is the -SH group.

3.3. Pb(II) stabilization tests using polyethyleneimine-derived dithiocarbamates (PDTCs)

3.3.1. Effect of chelating agent dosage on dissolved Pb(II) removal

Many previous studies have compared the chelation performance of heavy metals using different mass concentrations of chelating agents. However, chelating agents differ not only in molecular structure but also in sulfur content. A chelating agent with a higher sulfur content will have more dithiocarboxyl groups at the same mass concentration. Therefore, evaluating chelating performance solely based on different chelating agent concentrations does not account for the changes in properties due to variations in molecular weight.

To compare the dissolved Pb(II) removal efficiency of PDTCs, the amount added was based on elemental analysis results. As illustrated in Fig. 3a, when the molar ratio of -CSS- to Pb^{2+} was 2:1, nearly all dissolved Pb(II) was removed, consistent with the molar ratio of PDTC to CS_2. For molar ratios of less than 2:1, low molecular weight PDTCs displayed a better removal efficiency. It is well known that the position of nitrogen and sulfur atoms, the types of substituents, and other constituent atoms in dithiocarbamates significantly affect their performance. These results suggest that sulfur-containing -CSS- groups contributed effectively to Pb(II) chelation under the tested conditions.

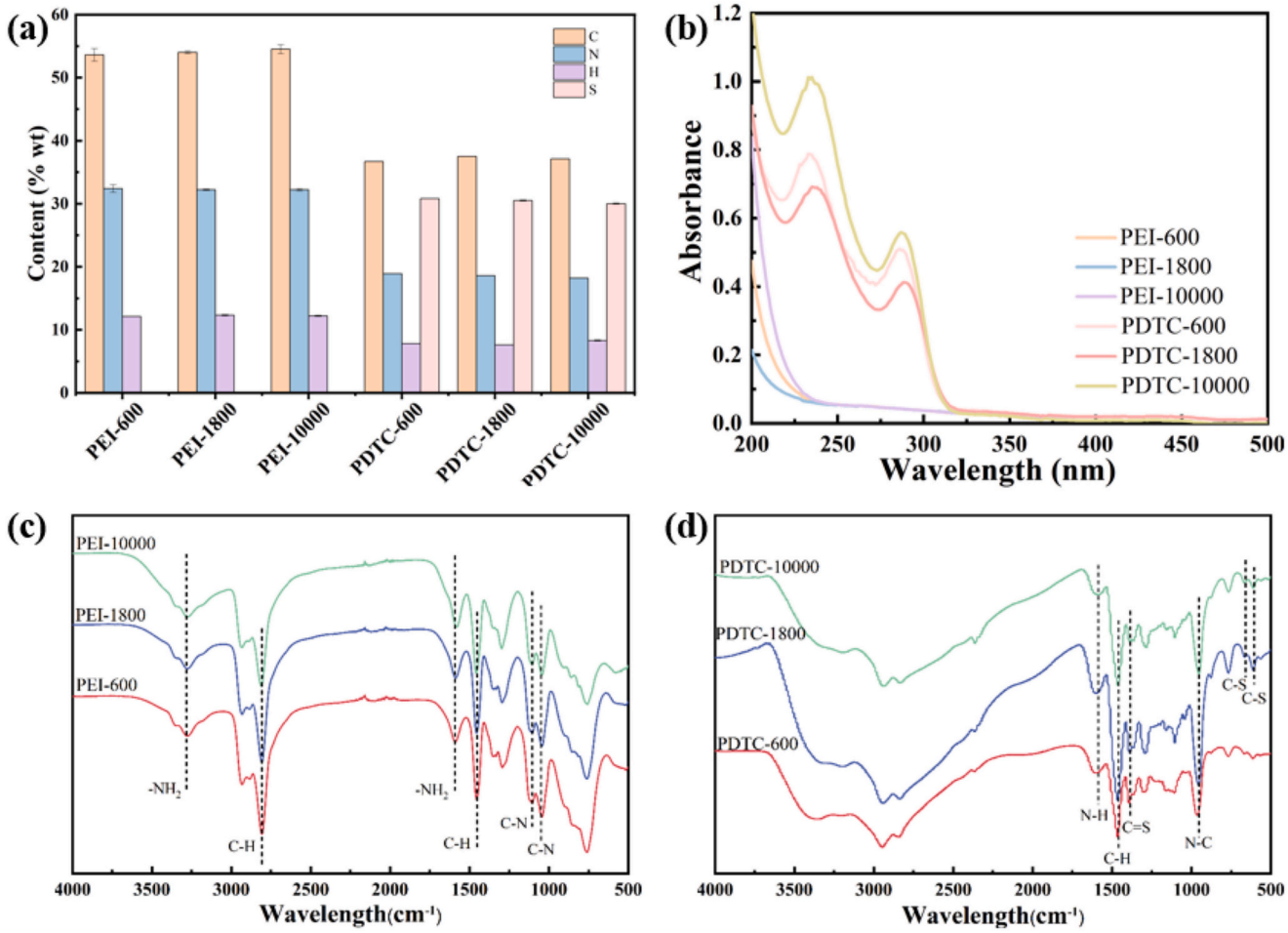

Fig. 2. Characterization of PDTCs: a) element analysis, b) UV–Vis absorption spectra. FT-IR spectra of (c) PEIs and (d) PDTCs.

Therefore, there is a need to produce chelating agents with a high sulfur content. SDD was used as a commercial dithiocarbamate benchmark, and PDTC-600 showed comparable or better Pb(II) stabilization efficiency under the tested conditions. Compared with sulfide-based precipitants, PDTCs may reduce concerns associated with H_2S release and sulfide oxidation.

3.3.2. *Effect of coexisting heavy metal ions*

Under a constant chelator dosage (molar ratio of S to Pb of 4:1), the removal efficiency of Pb^{2+} was investigated in the presence of different concentrations of coexisting heavy metals (Cu^{2+}, Zn^{2+}, Cd^{2+}, and Ni^{2+}).

As shown in Figs. 3b–e, the presence of coexisting heavy metals decreased the Pb(II) removal efficiency of PDTCs to different extents. With increasing concentrations of Cu(II), Cd(II), and Ni(II), more -CSS- sites were consumed by coexisting metals, resulting in lower Pb(II) stabilization efficiency. Overall, the inhibitory effect followed the order Cu(II) > Cd(II) > Ni(II) > Zn(II). This inhibition was mainly caused by competitive chelation, as these metals can consume available -CSS- groups and form metal–PDTC complexes, thereby reducing the active sites available for Pb(II). The stronger inhibition by Cu(II) may be related to its high affinity toward sulfur-containing ligands and the stability of Cu–PDTC complexes. Further experiments using real fly ash leachate or leachate-derived metal ratios are needed to confirm the competitive stabilization behavior under practical conditions.

3.3.3. *Effect of coexisting macronutrient metal ions*

Fly ash contains numerous macronutrient metals, such as Na, K, Ca, and Mg, in addition to heavy metals. These elements may impact the chelation-based stabilization of Pb(II) by the chelating agent. Under a constant chelator dosage (molar ratio of S to Pb of 4:1), the change in Pb^{2+} removal efficiency was investigated in the presence of varying concentrations of macronutrient metal ions (Na^+, K^+, Ca^{2+}, and Mg^{2+}) in the initial solution.

As illustrated in Figs. 3f–i, macronutrient metal ions affected the apparent Pb(II) removal efficiency of PDTCs to different extents. The Pb (II) removal efficiency of PDTC-10000 remained relatively stable within the tested concentration range, whereas PDTC-600 showed a decreasing trend as the concentration of Na^+, K^+, Ca^{2+}, or Mg^{2+} increased. This decrease may be related to changes in floc aggregation and settling behavior under high ionic-strength conditions. At elevated concentrations, coexisting inorganic cations may compress the electric double layer and alter the surface charge of Pb-PDTC precipitates, which can hinder effective coagulation and settling. Therefore, the reduced apparent removal efficiency of PDTC-600 in the presence of high macronutrient ion concentrations should be interpreted mainly as a settling/aggregation effect rather than a loss of intrinsic Pb(II) chelation ability.

When selecting a suitable chelating agent for Pb(II) stabilization in fly ash leachate-related systems, the impact of macronutrient metals on the chelation performance should be considered. Pre-treatment measures to remove these metals may be necessary to improve the chelation rate of the target heavy metals. In addition to cations, real MSWI fly ash leachate may contain dissolved organic matter and abundant anions such as Cl^-, SO_4^{2-}, and CO_3^{2-}, which may affect Pb(II) stabilization by changing Pb speciation, increasing ionic strength, or competing with PDTCs for metal binding.

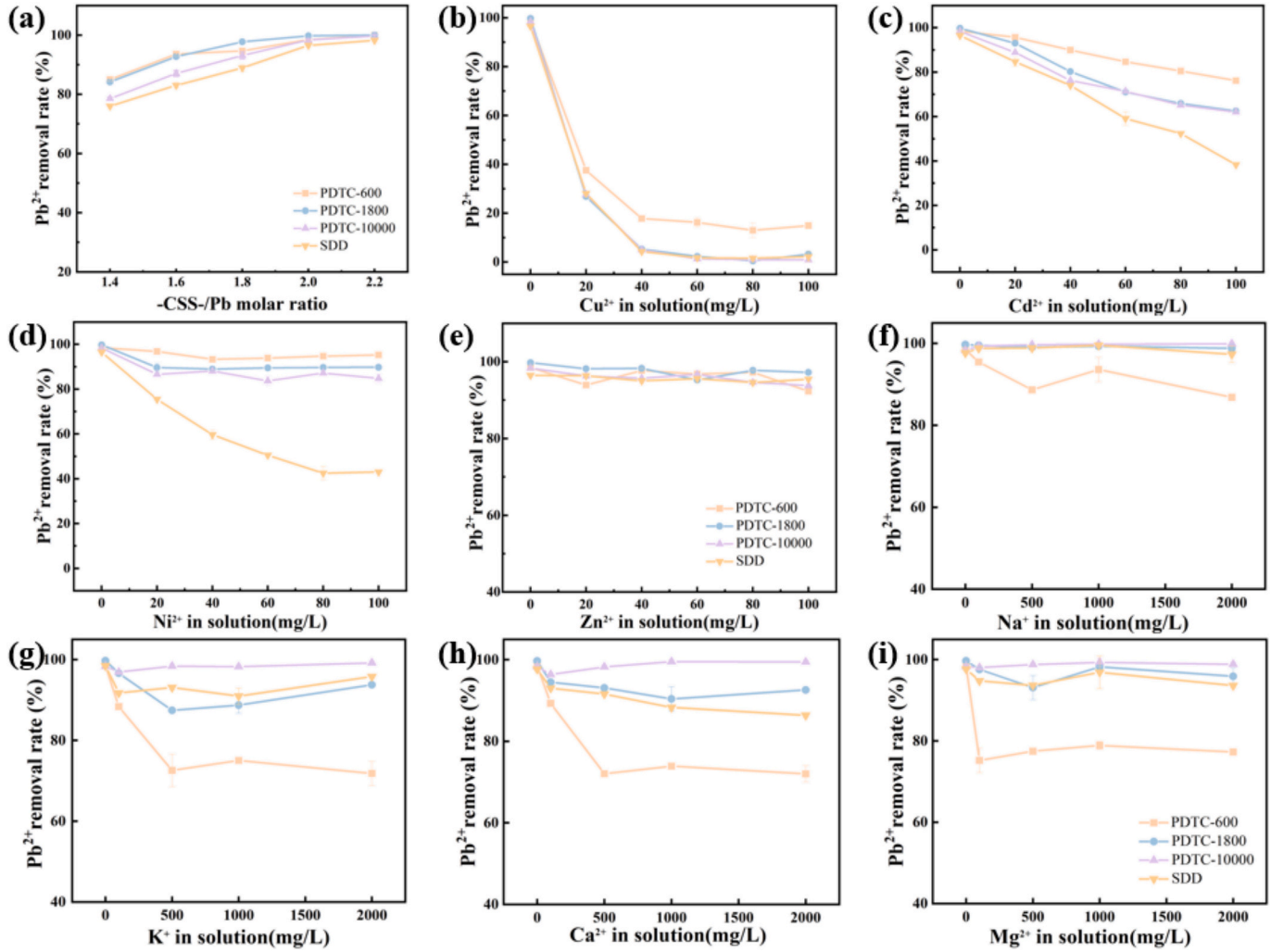

Fig. 3. Effect of a) chelating agent dosage on Pb removal rate on PDTCs. Effect of coexisting heavy metal ions, including (b) Cu^{2+} (c) Cd^{2+} (d) Ni^{2+} (e) Zn^{2+} on Pb(II) removal efficiency. Effect of common ions, including (f) Na^{+} (g) K^{+} (h) Ca^{2+} (i) Mg^{2+} on Pb(II) removal efficiency. Standard deviations were calculated based on triplicate experiments.

3.3.4. Pb stabilization mechanisms

After washing, drying, and grinding, the dithiocarbamate precipitates appeared as insoluble brown solids and were observed under a Hitachi Se3400N II scanning electron microscope. As illustrated in Fig. 4, PDTC-600-Pb appeared as flakes, PDTC-1800-Pb as lumps, and PDTC-10000-Pb as granules. The longer the carbon chain of the dithiocarbamate, the more carbon it contains. The FTIR spectra of PDTC-600-Pb and its corresponding precipitates (Fig.4a), PDTC-1800-Pb and its corresponding precipitates (Fig.4b), and PDTC-10000-Pb and its corresponding precipitates (Fig.4c) were analyzed. A higher metal removal efficiency was observed when the chelate product was more compact. Thus, the Pb removal efficiency was higher with PDTC-600 compared to PDTC-1800, and higher with PDTC-1800 compared to PDTC-10000.

To analyze the chelation mechanism, metal chelates generated by PDTCs of different molecular weights with Pb were collected for FT-IR analysis and compared with the corresponding PDTCs. The results are presented in **Fig. 5a** and **b**. Compared to the FT-IR spectra of PDTCs, the spectra of the three metal chelates (PDTC-Pb) display significantly weakened peaks from 700 cm^{-1} to 600 cm^{-1}, probably due to sulfur atoms in the dithiocarboxyl groups forming coordination bonds with Pb. Additionally, the stretching vibration of the N—C bond at 940 cm^{-1} for thioamide (N-C=S) was significantly weakened, while the stretching vibration peak of the C=S bond at 1390 cm^{-1} was significantly strengthened. These changes indicate that Pb^{2+} can chelate with the dithiocarboxyl groups on the PDTC molecular chains.

To further compare the interaction mechanisms of polyethyleneimine chelators with different molecular weights and Pb, the valence changes of elemental S in PDTCs and PDTC-Pb chelates were analyzed using XPS high-definition scanning (Fig. 6). The S2p energy spectra were found to be similar for the three different molecular weight chelators. For instance, PDTC-600 shows S2p peaks at 160.8 eV and 161.8 eV, corresponding to C—S and C=S bonds, respectively. The binding energies and areas of these bonds are consistent for the three chelating agents, indicating that molecular weight changes have little effect on the electronegativity of sulfur atoms, with the carbon–sulfur bonds predominantly being double bonds.

However, in the PDTC-Pb chelates, the S2p spectra indicate that the carbon-sulfur bonds are primarily single bonds. Specifically, in PDTC-10000-Pb, the binding energies of C—S and C=S bonds increase significantly compared to PDTC-10000. These shifts in S2p binding energy suggest that molecular weight may influence the local chemical environment of sulfur-containing groups in PDTCs. This change could affect the accessibility and coordination behavior of -CSS- groups toward Pb(II), although further spectroscopic and theoretical studies are needed to fully confirm this interpretation.

Due to the large radius and negative charge of sulfur atoms in dithiocarbamates, they are easily polarized and deformed under a

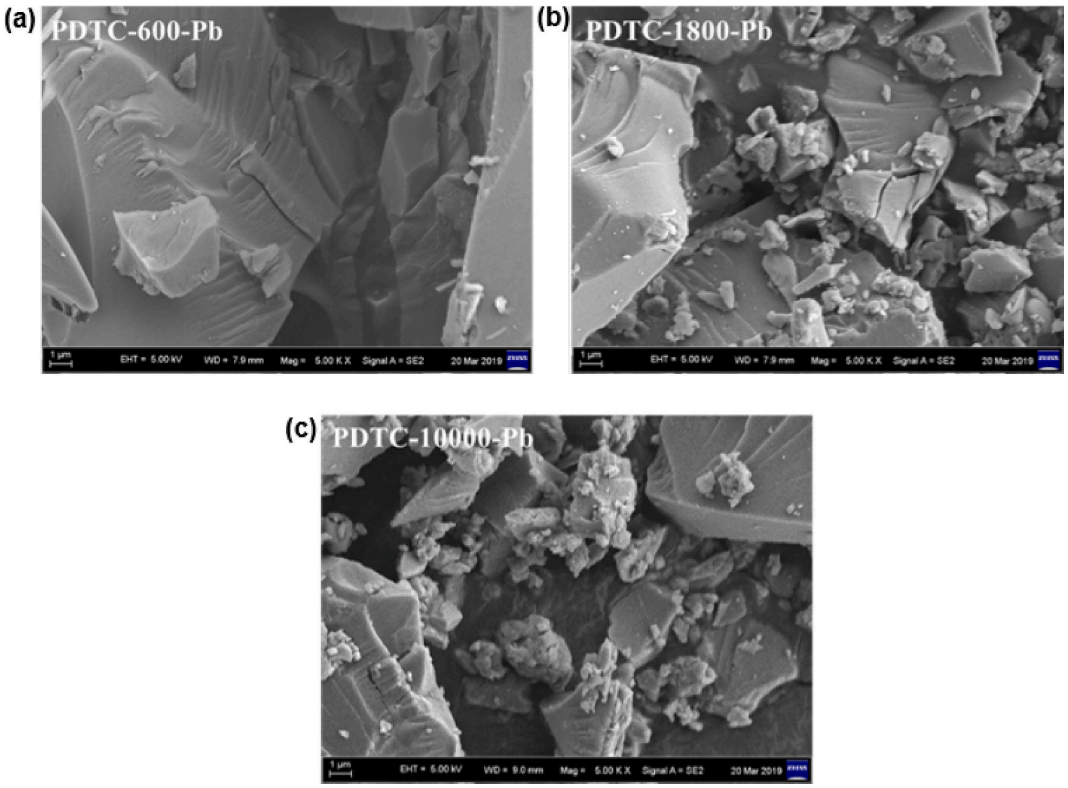

Fig. 4. SEM micrographs of different PDTC-Pb precipitates: (a) PDTC-600-Pb, (b) PDTC-1800-Pb, (c) PDTC-10000-Pb.

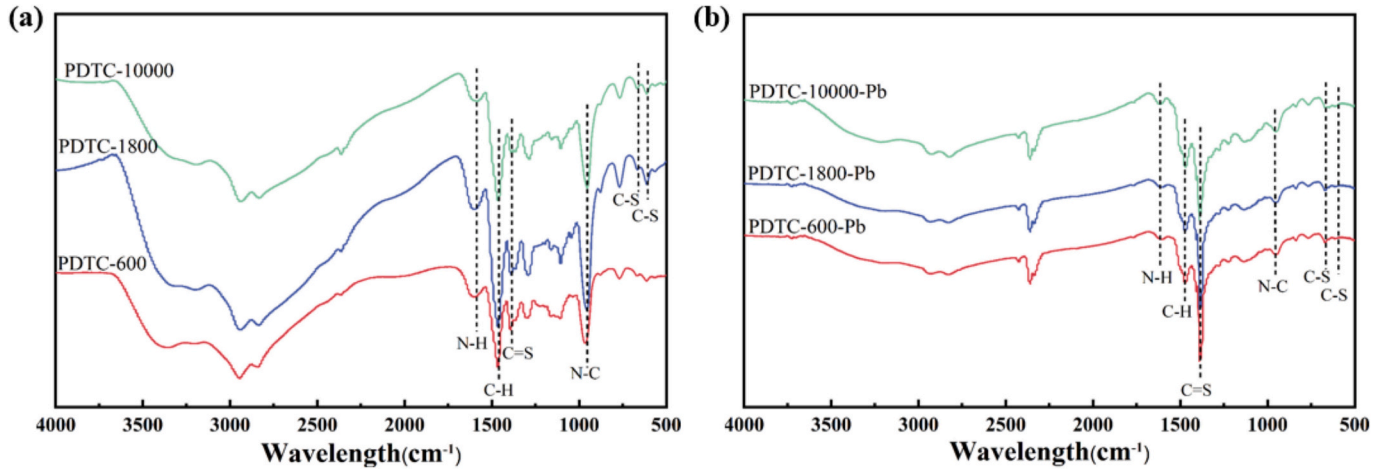

Fig. 5. FT-IR spectra of (a) PDTCs and (b) PDTC-Pb chelates.

negative electric field, causing SH groups to lose H^+, capture Pb^{2+}, and form insoluble chelates. Additionally, a weak blueshift makes the SH group less stable. As illustrated in Fig. 5, the FT-IR spectrum shifts to shorter wavelengths as the number of carbon atoms increases. This is due to increased steric hindrance and decreased conjugation with more carbon atoms, causing the molecular excitation wavelength to shift to shorter wavelengths. For example, the SH vibrational absorption peaks are at 1624 cm^{-1} for PDTC-600, 1672 cm^{-1} for PDTC-1800, and 1675 cm^{-1} for PDTC-10000. Based on the Pb(II) removal results and spectroscopic evidence, the apparent chelation performance under the tested conditions followed the order PDTC-600 > PDTC-1800 > PDTC-10000, which explains why PDTC-600 outperforms PDTC-1800 in lead removal, and PDTC-1800 outperforms PDTC-10000.

3.4. Stability assessment

The pH-dependent leaching behavior of Pb-PDTC precipitates was used to evaluate the stabilization performance of PDTCs, since effective stabilization should not only decrease dissolved Pb(II) concentration but also limit the remobilization of Pb from the formed precipitates.

3.4.1. Thermal stability analysis

The thermogravimetric analysis (TGA) and differential thermogravimetric (DTG) curves of PDTC-Pb and SDD-Pb chelates are presented in Fig. 7. The SDD-Pb chelate exhibits a single weight-loss phase (300–350 °C). In contrast, the PEI-based Pb–PDTC precipitates showed two main weight-loss regions. The first region at 150–170 °C may be associated with the decomposition of dithiocarbamate-related moieties and

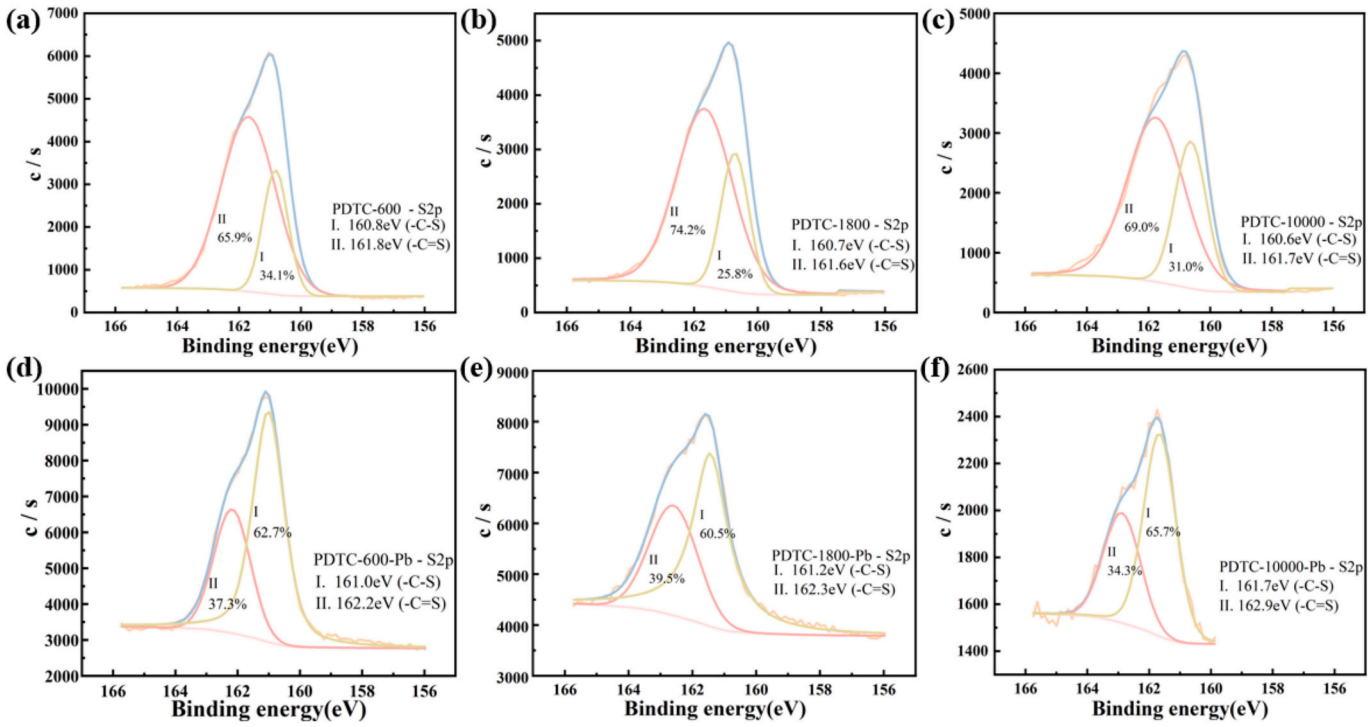

Fig. 6. High-resolution XPS spectra of S2p of PDTCs (a-c) and PDTC-Pb chelates (d-f).

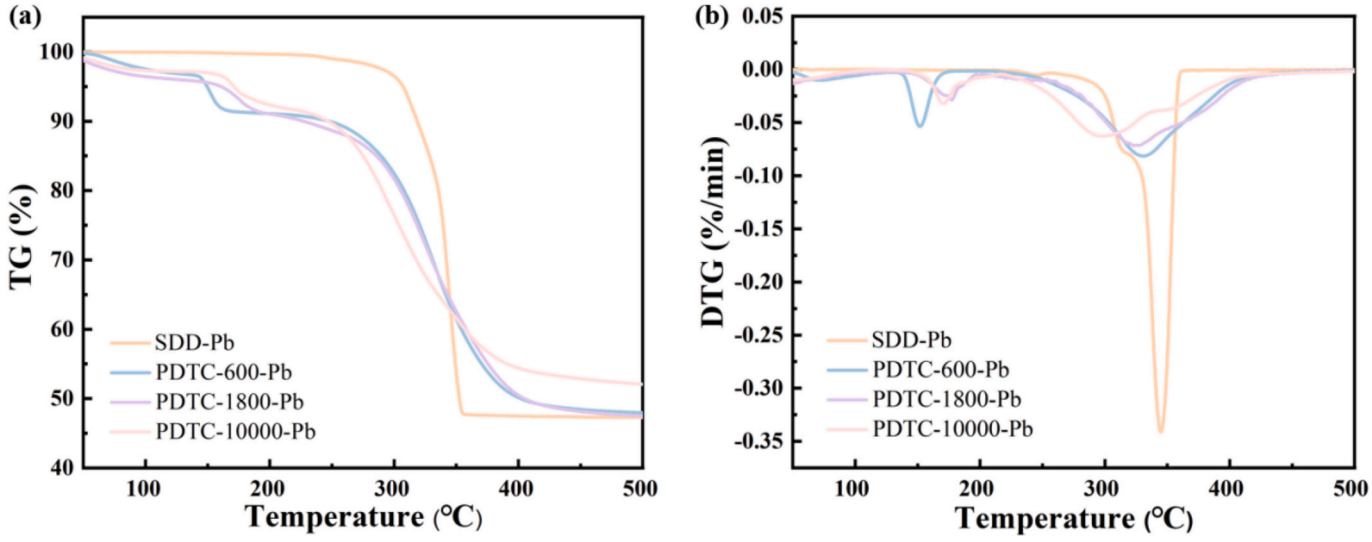

Fig. 7. Comparison of (a) TG curves and (b) DTG curves of PDTC-Pb and SDD-Pb chelates.

metal–sulfur coordination structures [44], while the second region at 300–350 °C may correspond to further degradation of the PEI-based polymer framework. The single weight-loss phase for the SDD-Pb chelate at 350 °C is probably due to the decomposition of the dithiocarboxylate group. Although the pyrolysis temperatures of the polymer chelator chelates are lower than those of the commercial chelator, they remain higher than typical ambient temperatures in domestic landfills [45].

3.4.2. *Acid stability of chelator precipitation*

The acid–base stability of PDTC-Pb chelates and SDD-Pb was investigated under different pH conditions, considering potential influences from waste leachate and rainfall. The metal dissolution rate of the chelates was calculated based on the concentration of dissolved metals.

As illustrated in Fig. 8, the dissolution rate of heavy metals in different chelates varied according to the solution pH and exposure time. The Pb dissolution rate decreased with increasing pH. At pH levels above 9, the metal chelates displayed good environmental stability with negligible dissolution. Under acidic conditions, Pb was dissolved from all types of chelates. Over time, the dissolution rate of heavy metals increased at different pH levels. For instance, in a solution at pH 5.6, the Pb dissolution rate for PDTC-600-Pb rose from 0.78% on day 1 to 2.84% on day 70, whereas for PDTC-10000-Pb, it increased from 15.1% to 77.8% over the same period. Higher-molecular-weight PDTCs showed greater Pb leaching under acidic conditions, probably because steric hindrance reduced the accessibility and effective coordination of -CSS- groups in the resulting precipitates. Overall, the acid–base stability of PDTC-600-Pb was more comparable to that of SDD-Pb (See Fig. 9).

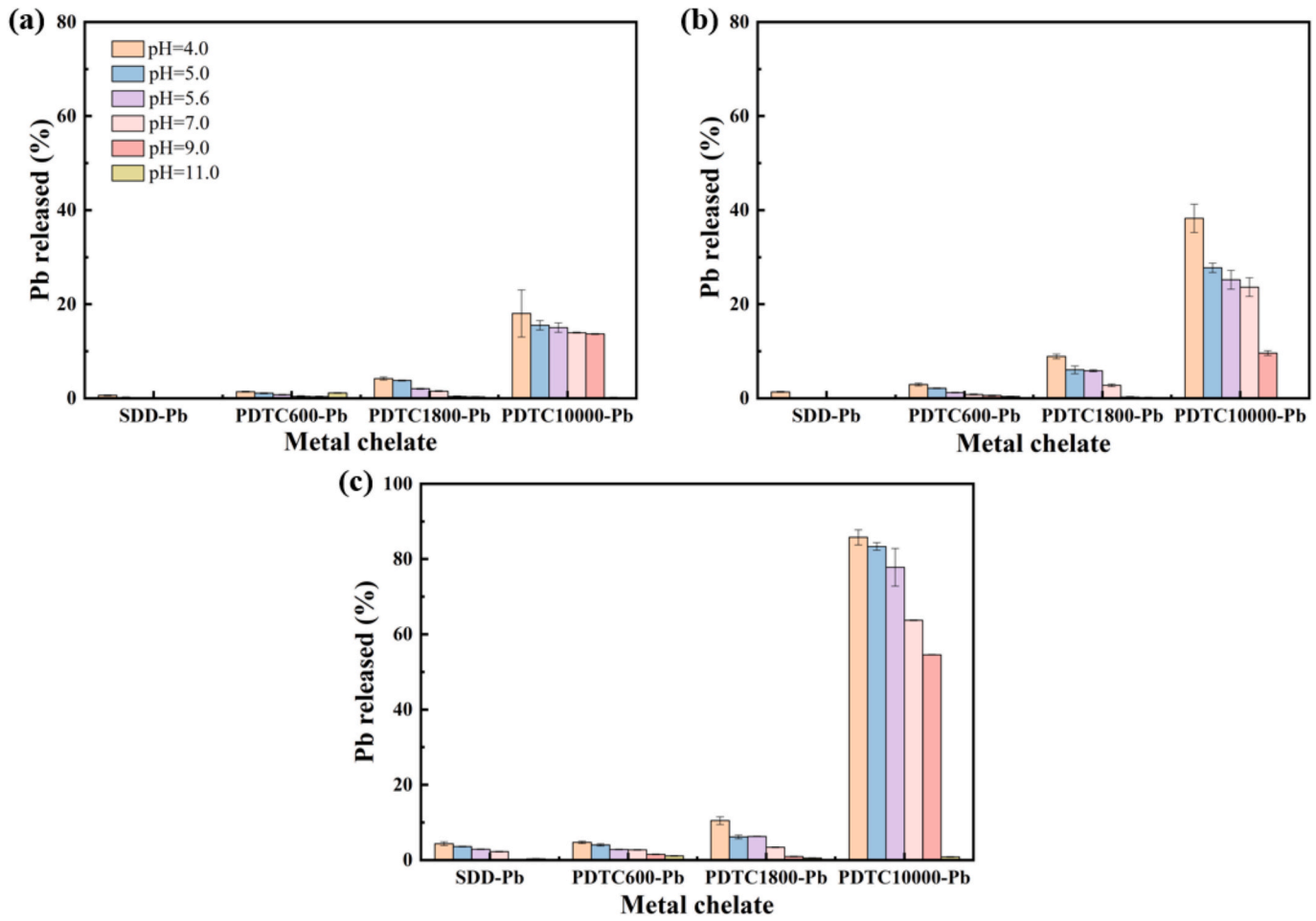

Fig. 8. Pb leaching rate of SDD-Pb and PDTC-Pb chelates under different pH conditions with various leaching times: (a) 1 d, (b) 5 d, (c) 70 d.

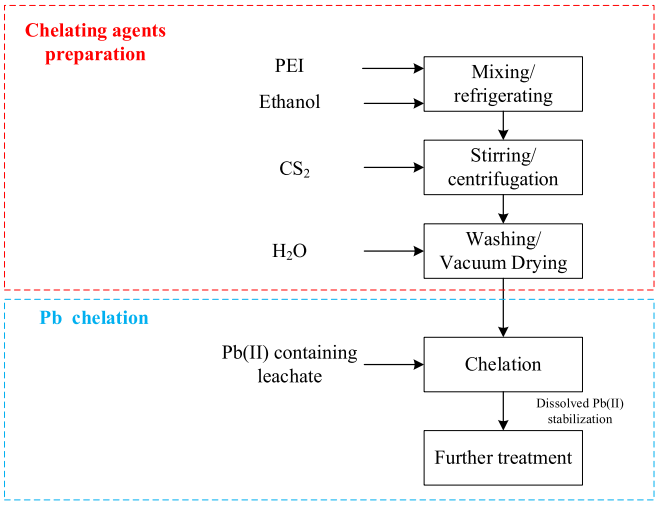

Fig. 9. Schematic diagram of PDTC synthesis and chelation-based Pb(II) stabilization in MSWI fly ash leachate-related systems.

Among the tested PDTCs, PDTC-600 exhibited the most favorable Pb (II) stabilization performance, with high dissolved Pb(II) removal efficiency and relatively low Pb leaching from the resulting Pb–PDTC precipitates under acidic conditions. This suggests that a lower molecular weight may improve the accessibility of -CSS- groups and facilitate Pb (II) chelation under the tested conditions.

It should be noted that fresh MSWI fly ash leachate can be more alkaline than the tested pH range; therefore, further validation under real highly alkaline leachate conditions is needed.

3.5. *Preliminary evaluation of potential economic and correlative impacts*

Economic evaluation accounts for both chemical and energy consumption, providing a thorough cost assessment. Based on the available data as presented in Table 2, the estimated cost per batch of experiments is approximately US$0.826, resulting in a treatment cost of approximately US$82.6 per ton of Pb(II)-contaminated fly ash, which is significantly lower than the cost of hazardous landfilling at

Table 2
Experimental inputs, energy consumption and waste products from one batch (10.0 kg MSWI fly ash).

Materials	Flow per batch	Cost (US$ per ton, market price in China, Sep. 2024)	Cost (US$ per batch)
PEI (g)	110	1390	0.152
Water for washing (L)	20	0.59	1.18×10^{-2}
Ethanol (L)	0.5	840	0.136
CS_2 (L)	0.153	700	0.133
Electricity		0.15/kWh	
Mixing (kWh)	1.2		0.18
Washing (kWh)	0.2		0.03
Vacuum drying (kWh)	0.5		0.075
Chelation (kWh)	0.8		0.12
Total			0.826

approximately US$240 per ton [46]. Notably, ethanol can be reused, and the novel synthesis method operates at room temperature, significantly reducing energy consumption and enhancing cost efficiency. Furthermore, scaling up from the lab batch to the pilot scale and then the industrial scale could further reduce both energy consumption and overall costs. This estimate should be regarded as a preliminary laboratory-scale cost assessment rather than a full engineering economic evaluation. Costs related to leachate generation, wastewater treatment, Pb-PDTC precipitate disposal, solvent recovery, labor, equipment depreciation, and scale-up were not fully included. Therefore, the comparison with hazardous landfilling cost is provided only as a reference.

The preliminary economic evaluation suggests that PDTC-based Pb (II) stabilization may have potential cost advantages compared with direct hazardous landfilling. However, this assessment remains preliminary and should be further validated at larger scales. It should also be noted that although the synthesis was conducted under mild NaOH-free conditions, CS_2 toxicity, ethanol recovery, washing, and drying should be considered in future process optimization.

In the advancing field of environmental technology, particularly concerning the use of novel PDTC chelating agents for the stabilization of heavy metals in fly ash, a detailed evaluation of stakeholder impacts is essential. The implications for various entities are systematically analyzed in Table 3, underscoring the equilibrium between prospective advantages and potential challenges.

Environmental protection agencies are projected to observe enhancements in environmental protection outcomes and a reduction in heavy metal pollution, as indicated in Table 3. This development necessitates modifications in regulatory frameworks and standards to accommodate the integration of new technologies. Government departments are anticipated to demonstrate a commitment to environmental preservation and public health. This progression, however, may require augmented financial support and adjustments in policy for seamless assimilation and governance of these novel agents. Enhanced efficiency in waste treatment and an improved environmental profile are expected outcomes for waste incineration plants. These benefits are accompanied by the necessity for elevated initial investments and operational expenditures, essential for the adoption of innovative technologies. For chelating agent manufacturers, Table 3 highlights the prospect of expanded market opportunities and the stimulation of technological innovation. Nonetheless, these entities are confronted with considerable research and development costs and market acceptance uncertainties. Research institutions and universities are identified as beneficiaries of new research avenues and scientific advancement. Concurrently, challenges in securing research funding and overcoming technical obstacles are prevalent in such pioneering endeavors. Communities and the public are likely to experience improved environmental quality and decreased health risks. However, a degree of uncertainty, particularly regarding the operational risks associated with new technologies, may initially provoke public skepticism or apprehension.

Table 3
Positive and negative impacts of employing novel PDTC chelating agents in the heavy metal stabilization of MSWI fly ash by potential targets.

Potential targets	Pros	Cons
Communities and the public	1. Improved environmental quality 2. Reduced health risks	1. Uncertainty. For instance, potential operational risks associated with new technology
Government departments	1. Demonstrating commitment to environmental protection 2. Improving public health standards	1. Additional financial support and policy adjustments
Incineration plants	1. Enhanced efficiency in waste treatment 2. Improved environmental image	1. Increased initial investment 2. Operational costs
Chelating agent manufacturers	1. Increased market opportunities 2. Driving technological innovation	1. High research and development costs 2. Uncertainty in market acceptance
Research institutions and universities	1. New areas of research 2. Advancement in science and technology	1. Facing challenges in research funding and technical hurdles
Environmental protection agencies	1. Improved environmental protection outcomes 2. Reduction in heavy metal pollution	1. Update regulatory frameworks and standards

In summary, the introduction of PDTC chelating agents in the treatment of MSWI fly ash signifies a notable advancement in environmental management. Nevertheless, this innovation also introduces a complex spectrum of impacts across various stakeholders. These impacts span regulatory, financial, technological, and perceptual dimensions, necessitating a thorough and balanced approach to fully leverage the potential of this technological development.

4. Conclusion

This study demonstrated that polyethyleneimine-derived dithiocarbamates (PDTCs) can effectively decrease dissolved Pb(II) concentration through chelation and precipitation, thereby achieving Pb(II) stabilization in MSWI fly ash leachate-related systems. The synthesis was conducted in ethanol under mild conditions without NaOH addition.

The molecular weight of PEI significantly affected the chelation efficiency and environmental stability of PDTCs. Among the tested agents, PDTC-600 exhibited the most favorable Pb(II) stabilization performance, suggesting that a lower molecular weight may improve the accessibility of active -CSS- groups. A higher molecular weight did not necessarily enhance stabilization performance, as increased steric hindrance may reduce Pb(II) chelation efficiency and acid stability. FT-IR and XPS analyses confirmed that sulfur-containing dithiocarbamate groups played a key role in Pb(II) binding and Pb–PDTC precipitate formation.

These findings provide guidance for designing polymeric dithiocarbamate chelating agents for Pb(II) stabilization in MSWI fly ash leachate. Future work should further verify the performance of PDTCs in real, highly alkaline fly ash leachate and in raw or pretreated fly ash systems, while optimizing chelator molecular weight to balance stabilization efficiency, precipitate stability, reagent safety, and environmental compatibility.

CRediT authorship contribution statement

Jinfeng Tang: Writing – original draft, Supervision, Resources, Funding acquisition, Conceptualization. **Yanyan Li:** Methodology, Data curation. **Xinmei Lin:** Investigation, Formal analysis. **Minhua Su:** Writing – review & editing. **Lizhi Tong:** Writing – original draft, Methodology, Formal analysis, Data curation. **Yun Zhu:** Writing – review & editing, Data curation. **Biming Chen:** Writing – review & editing. **Małgorzata Szlachta:** Writing – review & editing. **Kaimin Shih:** Writing – review & editing, Methodology. **Junhua Xu:** Writing – review & editing, Methodology, Investigation.

Declaration of generative AI and AI-assisted technologies in the writing process

During the preparation of this work the author(s) used ChatGPT in order to refine the English language. After using this tool/service, the author(s) reviewed and edited the content as needed and take(s) full responsibility for the content of the published article.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

Supplementary data to this article can be found online at https://doi.org/10.1016/j.seppur.2026.139713.

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