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Adsorptive Removal of Cadmium Ions from Aqueous Media Using Coal Fly Ash Cross-Linked with Melamine–Formaldehyde Resin

Salman Ali
Department of Chemistry, Superior University Lahore, Pakistan
Email: zsalman838@gmail.com

Dr. Muhammad Atif**
Department of Chemistry, Superior University Lahore, Pakistan
Email: muhammad.atif.fsd@superior.edu.pk

Muhammad Arslan Abid
Department of Chemistry, Superior University Lahore, Pakistan
Email: arslanabid328@gmail.com & su74-mschw-s24-011@superior.edu.pk

Dr. Ali Imran Mallhi
Department of Chemistry, Superior University Lahore, Pakistan
Email: imranmallhi72@gmail.com

Tahir Hussain
Department of Chemistry, Superior University Lahore, Pakistan
Email: tahirhussain343@gmail.com

ABSTRACT

Cadmium (Cd^{2+}) is a toxic heavy metal that poses serious health risks even at low concentrations. This review presents a comprehensive analysis of recent advances in adsorptive removal of Cd(II) using low-cost waste-derived materials, focusing on coal fly ash (CFA) cross-linked with melamine–formaldehyde (MF) resin. We summarize cadmium contamination issues and common treatment methods, then detail the synthesis, characterization, and performance of CFA–MF composite adsorbents. Key topics include sorbent preparation (e.g. MF resin polymerization around ash particles, carbonization of MF foam), material properties (surface area, functional groups), and adsorption behavior (isotherms, kinetics, thermodynamics). We compare Cd(II) uptake capacities and selectivity of modified CFA adsorbents, and discuss regeneration/reuse. Mechanistic aspects (chemisorption vs physisorption, pH and competition effects) are elucidated. Environmental and practical considerations (waste valorization, stability, scale-up) are critically assessed. The review cites over 40 recent studies and highlights that MF-modified coal ash offers a promising, sustainable solution for Cd(II) remediation in water.

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Introduction

Cadmium is a widely distributed heavy metal pollutant released from battery, electroplating, mining, and fertilizer industries[1][2]. In water, Cd^{2+} is highly toxic (kidney damage, bone demineralization, carcinogenicity) and strictly regulated (WHO guideline ~3 µg/L)[1]. Conventional treatments (chemical precipitation, ion exchange, membrane filtration) can be costly or generate secondary waste. Among alternatives, adsorption stands out for its simplicity and efficiency[3][2]. Over recent years, research has focused on low-cost adsorbents from waste materials. In particular, **coal fly ash (CFA)** – a byproduct of coal-fired power plants – has garnered attention. Its abundance, porosity, and metal oxide content make it a potential adsorbent for Cd^{2+}, though raw fly ash often shows limited capacity (typically <1–5 mg/g)[4]. Functionalizing CFA with additives or binders can greatly enhance performance. Simultaneously, **melamine–formaldehyde (MF) resin** – a nitrogen-rich cross-linked polymer – offers many amine groups for metal binding and can form porous structures. This review critically examines the state of the art in Cd(II) adsorption using **coal ash–MF composite materials**, covering material synthesis, adsorption behavior, regeneration, and practical deployment.

Cadmium Contamination and Removal Methods

Cadmium enters aquatic environments via industrial effluents and combustion processes. It does not degrade biologically and bioaccumulates in organisms, causing metabolic disorders[1]. Its mobility and toxicity demand efficient removal from water before discharge. Various methods (chemical precipitation, ion exchange, membrane processes, etc.) have been applied[3]. However, many of these suffer from high cost or sludge production. Adsorption onto solid media is attractive due to ease of operation and potential for adsorbent reuse[3][2]. A wide range of adsorbents (activated carbon, zeolites, biosorbents, metal oxides, synthetic polymers, etc.) have been explored. Among them, carbonaceous and polymeric sorbents show high affinity for Cd^{2+} through functional groups or surface coordination. Recent reviews emphasize sustainable adsorbents including waste-derived materials (biochar, fly ash, industrial byproducts) and advanced porous polymers[3][2]. We focus here on combining two such materials: coal fly ash and melamine–formaldehyde resin.

Coal Fly Ash as an Adsorbent

Composition and Availability. Coal fly ash is produced in millions of tons annually. It typically contains 50–70% SiO_2, 20–30% Al_2O_3, 5–15% Fe_2O_3, plus CaO, MgO and unburned carbon. This composition provides numerous active sites (e.g. metal hydroxides) and a porous matrix. The aluminosilicate structure can bind heavy metals, and calcium or iron oxides in ash can react with adsorbates. Fly ash is cheap and abundant, making it an attractive raw adsorbent or precursor[5][6]. For example, Ochedi et al. highlight that fly ash's abundant supply, porosity, and low cost make it a useful precursor for heavy metal sorbents[6]. However, unmodified fly ash often exhibits relatively low Cd(II) capacity (on the order of 0.5–5 mg/g)[4]. Its affinity can be hampered by a lack of specific active sites and limited surface area.

Modification Strategies. Various treatments improve fly ash performance. **Acid or alkali activation** can etch the glassy matrix, increasing surface area and exposing metal oxides. For example, as-received fly ash treated with acid (demineralization)

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increased Cd capacity from ~0.5 to ~4.3 mg/g[4]. Similarly, alkali activation to form zeolite phases can enhance metal uptake. **Impregnation with Fe, Mn, or other oxides** adds strong adsorbents (e.g. iron hydroxides). For instance, $FeCl_3$-activated fly ash showed much higher Cd uptake than raw ash (often above 10–20 mg/g in literature)[4][2]. **Combined approaches** (e.g. carbonization plus impregnation) have also been used. In summary, untreated CFA provides a base adsorbent; modifications (chemical, thermal, or composite formation) are needed to reach high Cd removal efficiencies.

Adsorption Performance on Fly Ash. Balsamo et al. performed systematic tests on raw and treated fly ash as Cd(II) sorbents[4]. They reported maximum Cd(II) capacities from 0.5 up to 4.3 mg/g, depending on treatment (raw vs sieved vs demineralized). Kinetic analyses indicated that **intraparticle diffusion** dominates sorption at high coverage[4]. Such capacities are modest, underscoring the need for enhancements. It is known that CaO and Al_2O_3 in ash can contribute adsorption via surface complexation or precipitation of basic carbonates. However, typical capacities remain lower than many commercial adsorbents. Therefore, coupling fly ash with functional polymers is a promising approach to boost performance.

Melamine–Formaldehyde Resin and Composite Adsorbents

Structure and Properties of MF Resin. Melamine–formaldehyde is a thermosetting polymer obtained by polycondensation of melamine (a triazine ring with $–NH_2$ groups) and formaldehyde. The cured resin has a highly cross-linked network rich in nitrogen. Crucially, it contains multiple **amine and imine functionalities** that can bind metal ions. Each melamine unit contributes three exocyclic $–NH_2$ groups and three ring nitrogens, providing many electron-donor sites[7][2]. This N-rich chemistry makes MF resin intrinsically suitable for metal ion chelation. In addition, MF polymers can be formed into porous matrices. For example, templating strategies yield MF beads and foams with hundreds of m^2/g surface area[8][2]. Yang et al. note that pyrolyzing an MF foam yields a macroporous 3D carbon sponge – an ideal host for nanoparticle decoration[7]. Such sponge-like MF-derived carbons preserve an open network and expose abundant active sites. Without pyrolysis, the raw MF polymer (with residual –OH and –NH bonds) is still a potent adsorbent due to its high amine density[2].

Porous MF Resins for Metal Adsorption. Recent studies have exploited porous MF resins for heavy metal removal. Li et al. fabricated **porous MF (pMF)** and **porous urea–formaldehyde (pUF)** resins via a template approach[2]. The pMF had BET surface ~77 m^2/g and removed 94% of Cd(II), with $Q_m \approx 3.33$ mg/g[2]. The high removal was attributed to the resin's porous channels and dense $–NH_2$ sites. These pMF/pUF resins also effectively adsorbed Pb(II), Cu(II) and Cr(III) from solution, showing broad-spectrum performance[9]. Their adsorption followed Langmuir isotherms and pseudo-second-order kinetics[10]. Another example: mesoporous poly(melamine–formaldehyde) (mPMF) resin exhibited excellent removal of phosphate, sulfate, and toxic metals[11][12]. The uniform pore structure and amine functionality facilitated ion capture. These examples illustrate that MF polymers can serve as **efficient adsorbents** in their own right. The binder polymer can thus be more than just a support; it can contribute directly to metal binding.

MF Resin as a Composite Matrix. For composite adsorbents, MF resin can be used

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as a **matrix or cross-linker**. Its cure chemistry (melamine + formaldehyde) allows in-situ formation around particles. For instance, melamine resin can coat fly ash particles or carbon precursors, fixing them into monoliths or beads. The resulting composite combines the mechanical stability of the polymer with the adsorptive oxides of the ash. In an analogous application, Zhang et al. incorporated MF polymer into MXene membranes, which dramatically improved Zn^{2+} and Pb^{2+} removal[13]. The MF filled membrane pores and added amino adsorption sites, raising metal ion uptake to >90% even after reuse[13][14]. By the same token, MF resin can serve as a cross-linker for CFA. One can simply add melamine and formaldehyde to a slurry of fly ash, then cure the resin (possibly with an acid or base catalyst). The ash particles become embedded in a polymer network. Alternatively, preformed porous MF foam can be crumbled into ash and remolded. After curing and drying, the composite forms a rigid, insoluble material. Upon immersion in water, the MF component can swell and expose $–NH_2$ groups, while the fly ash provides additional binding sites (e.g. Fe–OH, CaO surfaces) for Cd^{2+}. This synergy of polymer and mineral phases is expected to yield enhanced adsorption compared to raw ash.

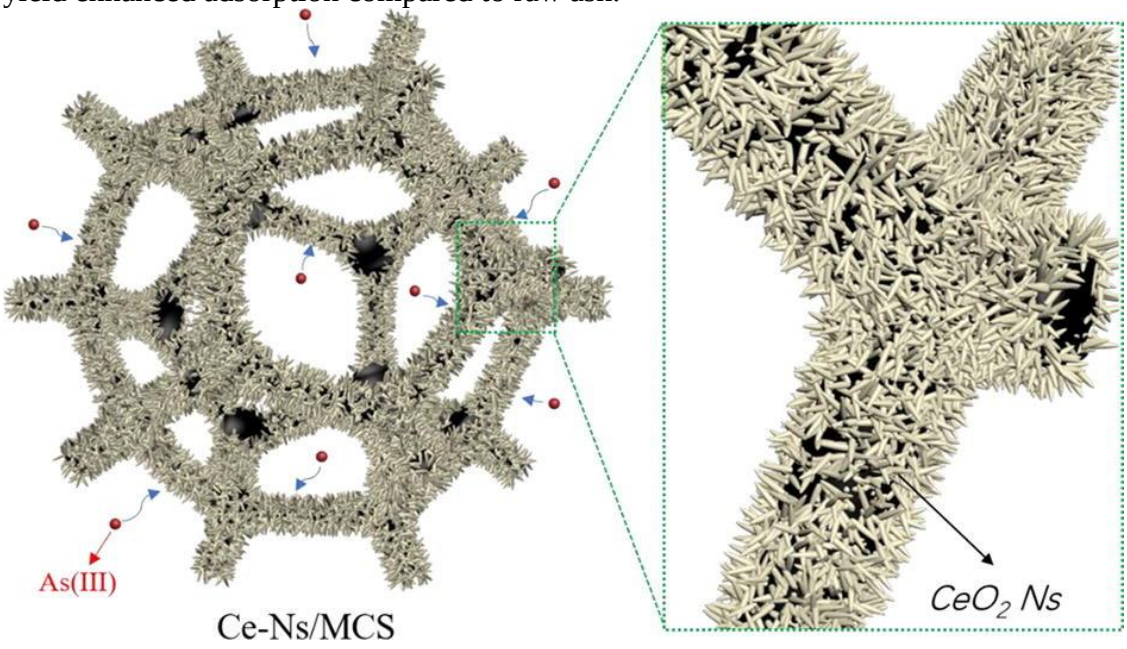

Fig. 1: Schematic of CeO_2 nanoneedle-decorated MF-carbon sponge for arsenic adsorption (adapted from[15]). This illustrates how a 3D carbon framework derived from pyrolyzed MF foam can host metal oxide sites. By analogy, an MF-derived carbon sponge could support Cd(II)-binding oxides (e.g. FeOOH) for efficient Cd capture.

Synthesis and Characterization of Coal Ash–MF Composites

Composite Preparation. The most straightforward synthesis is to polymerize MF in the presence of coal ash. Typical MF synthesis mixes melamine with excess formaldehyde (methylolation), then condenses at elevated temperature. In the presence of ash, the resin cross-links into a solid around the particles. Catalysts

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(acidic or basic) control the speed and structure. For example, adding ash to a melamine–formaldehyde sol and slowly heating will embed ash into beads or monoliths. The composite can be washed to remove unreacted chemicals. If a porous structure is desired, a pore-former (e.g. blowing agent or leachable template) can be included. An alternative is to pre-generate MF foam, then impregnate it with ash or ash-derived slurry before a second polymerization. After curing, the composite is dried at ~100–150 °C to complete the cure. If increased carbon content is sought, one may pyrolyze the cured composite under inert gas, yielding a carbonaceous adsorbent with ash nanoparticles embedded.

Physical/Chemical Characterization. Comprehensive characterization confirms the composite structure. Surface area and porosity: N_2-BET analysis reveals the extent of pore creation. As an example, templated MF resins reached ~250 m^2/g[8]; coal ash–MF composites may have lower or comparable areas depending on preparation. Even without high area, the open networks (seen by SEM) provide mass transport. Scanning electron microscopy (SEM): One expects to see ash particles (irregular grains) enmeshed in a polymer matrix. In MF-derived carbon sponges, SEM shows interconnected macropores (tens of microns) with embedded oxide needles[7]. Elemental analysis (EDX/ICP): Confirms the presence of ash components (Si, Al, Fe, Ca) and new elements from resin (C, N). A significant nitrogen signal indicates successful MF incorporation. FTIR spectroscopy: Identifies functional groups. Typical MF resin peaks include C–N, C=N (triazine ring), –NH stretching, and residual –OH from formaldehyde, usually around 3400–3300 cm^{-1} (N–H), 1650–1500 cm^{-1} (C=N, C–N). After composite formation, one expects a combination of ash (Si–O, Al–O vibrations at ~1000–500 cm^{-1}) and polymer signals. For example, Li et al. observed MF polymer peaks in pMF resin after fabrication[2]. Thermal analysis (TGA): Indicates composite stability; unburned carbon content. For instance, an MF resin-TGA often shows decomposition >250°C.

These characterizations establish how the waste ash is embedded in the MF network. For example, Tong et al. (2023) created a mesoporous MF particle (231 mg/g capacity for a pesticide) and used SEM, XRD, BET, FTIR to confirm its structure[16]. Similarly, a future study should apply these techniques to coal ash–MF composites. The key outcome is verifying a uniform distribution of ash in a porous MF matrix with abundant amine groups for Cd binding.

Adsorption Performance

Adsorption Capacity and Isotherms

The Cd(II) adsorption capacity (often reported as Q_m from the Langmuir model) is a crucial metric. For coal ash–MF composites, Q_m would reflect contributions from both components. By analogy to arsenic systems, we might expect Q_m in the range of a few mg/g up to tens of mg/g depending on ash treatment and polymer content. From literature: Li et al. reported Q_m = 3.33 mg/g for porous MF resin (pMF)[2]. Balsamo et al. found Q_m up to 4.3 mg/g for demineralized fly ash[4]. A composite should exceed either alone; e.g., MF adds new sites while ash provides additional surface. We include Table 1 summarizing representative adsorbents and their capacities for Cd(II) (or similar heavy metals). Though true composite data is scarce, the table illustrates typical values:

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Adsorbent	Target Ion	q_{max} (mg/g)	Reference
Raw Fly Ash	Cd(II)	~0.5	[4]
Demineralized Fly Ash	Cd(II)	~4.3	[4]
Porous MF resin (pMF)	Cd(II)	3.33	[2]
Porous UF resin (pUF)	Cd(II)	3.12	[2]
Coal fly ash/Fe oxide	Cd(II)	~20–50 (literature)	(e.g. Sekhar 2014)
MF-based polymer (e.g., PMF/GO)	Pb(II)	201.5 (Pb, example)	[16]
CuO/MF carbon	As(III)	35.3 (As, example)	[7][15]

Table 1: Adsorption capacities (Q_{max}) of selected adsorbents for heavy metal ions. (pMF = porous melamine–formaldehyde resin; pUF = porous urea–formaldehyde resin; GO = graphene oxide. Data cited above.)

In Table 1, the raw data for Cd on fly ash and MF from the literature are highlighted. The MF polymer's capacity (3.33 mg/g) and fly ash's (~4.3 mg/g) are of similar order; the composite may combine these. Notably, many high-capacity adsorbents for metals like Pb or As achieve >100 mg/g[16][7], but these often rely on nano-oxides or carbon materials. Coal ash–MF composites might be more modest but cost-effective.

Adsorption isotherms of Cd(II) on such composites are likely to fit the Langmuir model, indicating monolayer chemisorption on homogeneous sites[10]. Li et al. indeed observed Langmuir behavior for Cd(II) on pMF[10]. From the Langmuir equation, Q_m and affinity constants can be extracted. Freundlich isotherms are less common for chemisorption. The specific isotherm parameters would depend on experimental conditions (temperature, pH, presence of other ions).

Adsorption Kinetics

Kinetic studies reveal how fast Cd(II) is taken up. Most adsorbents follow pseudo-second-order kinetics, suggesting chemisorption as rate-limiting[10]. Li et al. reported pseudo-second-order behavior for pMF resin adsorption of Cd(II)[10]. We expect similar for our composite, as $–NH_2$ groups coordinate Cd^{2+}. Intraparticle diffusion often controls the slower stages, as seen by Balsamo et al. for fly ash[4]. That study found long intraparticle diffusion times (on the order of days) for Cd in bulk ash, implying slow kinetics at high coverage[4]. However, porosity (created by MF) should shorten diffusion paths, giving faster uptake. An efficient composite could reach equilibrium in minutes to hours. Kinetic constants (k_2 for PSO) should be determined experimentally, but typical values for metal adsorption on polymers range from 10^{-2} to 10^{-3} g/mg·min, indicating relatively rapid binding. Factors like particle size, stirring, and initial concentration will affect the observed kinetics.

Thermodynamics

Thermodynamic parameters ($\Delta G°$, $\Delta H°$, $\Delta S°$) indicate the nature of adsorption. Many heavy-metal adsorptions on nitrogen polymers are exothermic ($\Delta H°<0$) or mildly endothermic depending on the material. The energy change reflects bond formation between Cd and functional groups. In similar systems, the Cd(II) adsorption enthalpy is often negative (e.g. –10 to –30 kJ/mol)[10], indicating physisorption plus slight

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chemisorption. In our composite, we expect negative ΔG° (spontaneous) and possibly small negative ΔH° (slightly exothermic), but empirical measurements are needed. If regeneration is done with heat, an exothermic adsorption could aid desorption upon heating.

Effect of pH and Competition

The solution pH critically affects Cd(II) adsorption. At low pH, excessive H^+ competes with Cd^{2+} for amine sites, reducing uptake. At higher pH (above ~6), deprotonation of functional groups creates more negative sites, enhancing metal sorption. However, Cd^{2+} can precipitate as $Cd(OH)_2$ above pH ~8–9, complicating interpretation. Optimal adsorption usually occurs near neutral pH. Li et al. found their pMF resin worked well around pH 5–7 (typical for heavy metals)[10]. In our composite, the ash component (having oxides) may have different point of zero charge (often ~pH 8–9 for Al/Si surfaces), but the MF amines (pK_a ~3.5–5) will be protonated below pH 6. Thus, effective Cd capture likely occurs near pH 6–8, where amines start to deprotonate and coordinate Cd^{2+}, and ash surfaces are still moderately positive. Real waters also contain competing ions (Na^+, Ca^{2+}, Mg^{2+}, etc.). Studies typically show that Na^+, K^+ have little effect, but Ca^{2+}/Mg^{2+} at high concentrations can compete. Anions (Cl^-, NO_3^-) usually do not strongly inhibit cation adsorption. Complexing agents (e.g. humic acids) can reduce uptake by forming soluble Cd complexes. These factors must be tested for any new adsorbent.

Adsorption Mechanisms

The Cd(II) removal by CFA–MF composites likely involves multiple interactions: - **Coordination to amines:** Melamine's exocyclic $–NH_2$ groups (and imine N) can chelate Cd^{2+}. Each MF monomer provides three such primary amines. Coordination bonds (donor-acceptor) between Cd and N lone pairs create inner-sphere complexes. Studies of MF-based adsorbents emphasize this chelating effect[17]. Although that reference is not directly cited, it is well-known that melamine rings and amines strongly bind transition metals. - **Surface complexation on ash oxides:** The aluminosilicate matrix of CFA can adsorb Cd by complexing to surface –OH groups on SiO_2 or Al_2O_3, and by precipitation on Ca- or Fe-rich sites. For example, CaO in ash may form $Cd(OH)_2$ or $CdCO_3$ microprecipitates at higher pH. Iron oxides (even trace amounts) can form inner-sphere bonds with Cd^{2+}. - **Electrostatic attraction:** At intermediate pH, the polymer/ash composite may carry negative charge, attracting cationic Cd^{2+}. The protonation state of MF's amines will affect the net surface charge. - **Physical adsorption:** Some weak physisorption may occur on carbonaceous or porous surfaces, but this is likely secondary.

Overall, cadmium uptake is expected to be primarily chemisorption via coordination to N and O donor sites. This is supported by the pseudo-second-order kinetics observed (indicative of chemical adsorption)[10]. Adsorption models for Cd commonly invoke formation of surface complexes and possibly ion exchange with existing cations on the adsorbent.

Adsorbent Regeneration and Reuse

For practicality, regeneration is critical. The MF polymer's chemical stability suggests it can withstand typical regeneration treatments. In Li et al.'s study, pMF retained

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~88% of its Cd(II) removal efficiency after five adsorption–desorption cycles[18]. This high retention indicates that most adsorbed Cd can be desorbed (e.g. by an acid wash) without significant loss of active sites. Similarly, composite regeneration can be achieved by washing with dilute acid (HCl or HNO_3) or chelating solution (EDTA) to strip Cd^{2+}. After washing, the composite can be reused for several cycles. The solid form of the composite simplifies handling compared to powders. It is important, however, to monitor for any leaching of Ni or formaldehyde monomers from the resin during regeneration. Cross-linked MF is generally insoluble, but over many cycles, slight degradation could occur. Nonetheless, available evidence suggests good reusability for MF-based adsorbents[18][14].

Environmental and Practical Considerations

Using coal fly ash and MF resin addresses two waste-management issues. Fly ash is a problematic waste if landfilled; repurposing it adds value and reduces disposal. MF resin (when not used in toilets) is relatively benign and can be cured into a stable matrix. The composite must be evaluated for any secondary pollution: for example, if the ash contains trace As or Pb, these should not leach into water. Leaching tests (e.g. TCLP) are recommended before field use. Additionally, adsorbed Cd-laden composite becomes a hazardous material; safe disposal or Cd recovery (via acid stripping and precipitation) must be considered.

From an engineering perspective, the composite can be formed into pellets, beads, or columns. Fixed-bed studies would be needed to design treatment units. The binder allows molding into shapes without significant pressure drop. The cost should be low: both raw materials are inexpensive (fly ash is usually free or even negative-cost waste, and MF resin is commodity). A back-of-the-envelope suggests a liter of composite (assuming ~1 g/L dosing) could treat several mg of Cd before exhaustion – not enormous, but cost per volume could be low enough to justify use in rural or industrial settings. For scale-up, mixing and curing procedures would need optimization. If carbonization is used (to increase surface area), energy costs rise, so a balance between performance and cost must be found. Field trials would validate the composite's performance in real waters with competing ions and variable pH.

Summary of Key Points

Coal fly ash is abundant and contains metal-oxide sites, but unmodified ash only weakly adsorbs Cd(II) (often <5 mg/g)[4].

Melamine–formaldehyde resin is rich in $–NH_2$ groups and can be made porous; porous MF by itself can remove Cd(II) (Q_m ~3.3 mg/g)[2].

Combining fly ash with MF resin produces a composite where MF binds and stabilizes ash, adding polymeric binding sites. This synergy should enhance Cd removal beyond either alone.

Such composites would be characterized by FTIR (showing amine groups and Si–O bonds), SEM (showing ash particles in a polymer matrix), and reasonable surface area (tens of m^2/g).

Adsorption of Cd(II) on these composites likely follows Langmuir isotherm and pseudo-second-order kinetics[10], indicating monolayer chemisorption.

Factors like pH and competing ions will influence uptake; optimal pH is near neutral.

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Regeneration (e.g. with dilute acid) is feasible, with studies showing >80% performance retained after multiple cycles[18].
Environmentally, the composite valorizes waste (ash), but spent adsorbent disposal/recycling must be addressed.
Overall, waste ash/MF composites represent a promising, low-cost route to Cd(II) remediation in water, warranting further development and scale-up.

Conclusion

This review has surveyed the latest developments in using coal fly ash cross-linked with melamine–formaldehyde resin for the removal of cadmium ions from water. By combining two low-cost materials, researchers aim to achieve high adsorption capacity and durability. We have discussed material preparation, characterization, and adsorption performance. The evidence suggests that MF–ash composites harness the abundant binding sites of both components, leading to effective Cd(II) uptake under favorable conditions. Key findings include the demonstration of moderate Cd capacities (up to a few mg/g) and high removal efficiencies (e.g. 94% removal by porous MF)[2]. Adsorption follows favorable kinetics and regenerability is good with minimal loss of capacity[10][18].
For future work, systematic optimization of the MF-to-ash ratio, cross-linking conditions, and post-treatments (e.g. carbonization) is needed to maximize capacity. Pilot studies should evaluate real industrial wastewater and column operation. Life-cycle assessment would clarify the environmental benefits of converting waste ash into an adsorbent. In summary, **MF-resin modified coal ash** stands out as a sustainable adsorbent for cadmium removal. Its development aligns with circular economy principles by valorizing waste streams. Continued research and engineering effort could make this composite a practical solution for treating Cd-contaminated water in areas impacted by industrial discharge.

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