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Review article

Crosslinked polymer nanocomposites for wastewater heavy metal adsorption: A review

Chi Loon Ng[1], *Wen Shyang Chow*[1*], *Azam Taufik Mohd Din*[2], *Cheu Peng Leh*[3], *Suchart Siengchin*[4]

[1]School of Materials and Mineral Resources Engineering, Engineering Campus, Universiti Sains Malaysia, Nibong Tebal 14300 Penang, Malaysia
[2]School of Chemical Engineering, Engineering Campus, Universiti Sains Malaysia, Nibong Tebal 14300 Penang, Malaysia
[3]School of Industrial Technology, Universiti Sains Malaysia, Gelugor 11700 Penang, Malaysia
[4]King Mongkut's University of Technology North Bangkok 1518 Pracharaj 1, Wongsawang Road, Bangsue, 10800 Bangkok, Thailand

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Abstract. Public health concerns caused by heavy metals present in wastewater have come to global attention. Heavy metal contamination of water resources will have a negative impact on marine species, the environment, and the ecosystem. A variety of wastewater treatment methods have been developed and improvised throughout the decades to counter this issue. Among the wastewater treatment methods, the adsorption method is believed to have a high potential and is suitable for removing heavy metals from wastewater. This review focuses on selecting various types of crosslinked polymer nanocomposites (CPN) as having a significant role in the removal of heavy metals from wastewater via the adsorption method. The adsorption capacity of the CPN was influenced by the polymer types, nanofiller selection, synthesis methods, and exposure conditions. This article will give us important information to improve the functionality of CPN as an adsorbent in wastewater treatment. The challenge, SWOT (strengths, weaknesses, opportunities, and threats) analysis, and future aspects of the CPN are shared in this review.

Keywords: *nanocomposites, crosslinked polymer, wastewater, heavy metals, adsorption*

1. Introduction

Wastewater is polluted water discharged after the use of commercial, domestic, industrial, or agricultural activities. This discharged water is hazardous for further use as it contains different kinds of contamination [1]. Chemical and biological contaminants are the two broad categories of water pollution contaminants. Known chemical contaminants are heavy metals, nitrogenous compounds, and hydrocarbons. On the other hand, biological contaminants are various kinds of bacteria and viruses [2]. Heavy metal pollution from wastewater has attracted worldwide attention recently, along with rapid global development. Common sources of heavy metals are mainly the discharge of industrial (*e.g.*, mineral and metallurgical processes) and domestic and agricultural effluent into the rivers [3, 4].

Heavy metals are known as metallic elements, and their density is at least five times greater than the density of water. For instance, elements such as lead (Pb), cadmium (Cd), arsenic (As), mercury (Hg), and chromium (Cr) are examples of heavy metals [5]. Heavy metals can have several implications for the ecosystem. The accumulation of heavy metals in the

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[*]Corresponding author, e-mail: shyang@usm.my

water ecosystem affects marine life as well as human beings through the food chain. Heavy metals are extremely hazardous as they are mostly carcinogenic and damage the human organ systems [6, 7].

Every country promulgates its own regulations to monitor and control the heavy metal content of wastewater discharged by industries. For instance, in China, the discharge standard of pollutants for municipal wastewater treatment plants (GB 18918-2002) was formulated by the Ministry of Environmental Protection of the People's Republic of China (MEP) in 2002 [8]. On the other hand, effluent in Singapore is regulated under the Environmental Protection and Management (Trade Effluent) Regulations by the National Environment Agency (2008) [9]. As for Malaysia, the discharge of wastewater follows the Environmental Quality (Industrial Effluent) Regulations 2009, which were established by the Department of Environment Malaysia in 2009 [10]. Based on their respective assessments, various countries developed their own regulations. According to the stated regulations, guidelines are made to ensure the industries comply with the standard. Table 1 shows Malaysia's standard on the accepted limit of the metal content of industrial effluent discharged and its respective potential for human effects at high doses (*i.e.*, the heavy metal doses higher than the permitted level). Standard A is for any inland waters within certain catchment areas as specified, whereas Standard B is for any other inland waters or Malaysian waters. High doses of heavy metals could cause negative impacts on human beings, for example, kidney problems, brain damage, skin irritations, disturbed hormone levels, nausea, *etc.* [11–15].

There are plenty of techniques that have been developed for wastewater treatment, which are classified as physical, chemical, and biological approaches. Physical treatments include techniques such as adsorption, membrane filtration, and coagulation. As for chemical methods, they consist of chemical reactions such as oxidation and ozonation. Biological methods, instead, utilize enzymes and microorganisms. Different techniques demonstrate their own advantages and limitations. The selection depends on various factors such as cost, time, and wastewater composition [16]. In heavy metal removal, commonly used techniques are chemical precipitation, flotation, ion exchange, coagulation-flocculation,

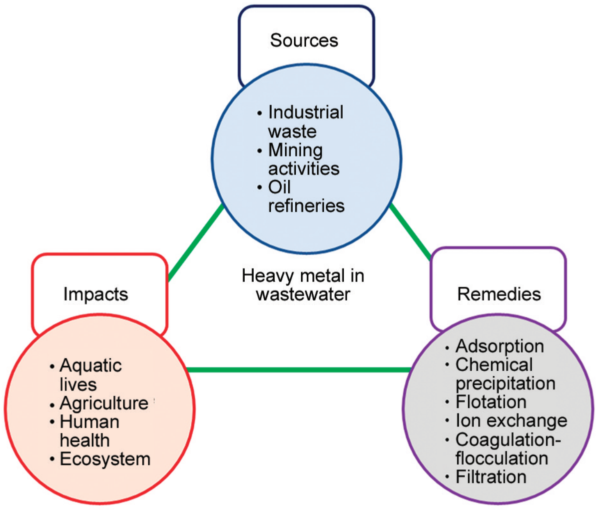

Figure 1. The impact of heavy metals on the environment and its remedies.

Table 1. Accepted limit for the discharge of industrial effluent and its respective potential effects.

Parameter	Unit	Standard A	Standard B	Negative effects of heavy metals	References
Mercury	[mg/l]	0.005	0.050	Kidney and brain damage, neurological disorder.	[11]
Cadmium	[mg/l]	0.01	0.02	Lungs and respiratory irritation, renal dysfunction and kidney disease.	
Arsenic	[mg/l]	0.50	0.10	Heart problem, brain damage, risk of cancer.	
Lead	[mg/l]	0.10	0.50	Affects gastrointestinal tract and central nervous system	
Manganese	[mg/l]	0.20	1.00	Negatively affect nervous system	
Chromium, Trivalent	[mg/l]	0.20	1.00	DNA damage, ulcer in nasal septum, damage to haemoglobin.	[12]
Chromium, Hexavalent	[mg/l]	0.05	0.05		
Aluminium	[mg/l]	10	15	Contact dermatitis, bone and brain damage	
Iron	[mg/l]	1.0	5.0	Formation of gastrointestinal ulcerations, risk of asbestosis	
Copper	[mg/l]	0.2	1.0	Disrupt respiration, damage in kidney and liver.	[13]
Nickel	[mg/l]	0.2	1.0	Cyanosis, bone nose and lung cancer	
Zinc	[mg/l]	2.0	2.0	Anaemia, nausea, skin irritations.	
Selenium	[mg/l]	0.02	0.50	Affects lipid profiles and altered thyroid hormones levels.	[14]
Silver	[mg/l]	0.1	1.0	Nausea, diarrheal and graying in skin	[15]

membrane filtration, and adsorption. Figure 1 depicts an illustration of the sources and effects of heavy metals in wastewater, as well as possible remedial measures.

The respective techniques' descriptions and some of their advantages and disadvantages are displayed in Table 2 [17, 18]. Of the methods mentioned, adsorption is known as a cost-effective, yet competent method compared to other heavy metal removal methods. The main advantage of this method is its ability to produce high-performance effluent as an end product. Moreover, this method is convenient to use and eco-friendly in that it does not produce toxic pollutants [19]. Accordingly, this review focuses on the adsorption method used by crosslinked polymer nanocomposites in the removal of heavy metals from wastewater resources.

The presence of heavy metals in various wastewater resource locations may cause health concerns in the surrounding community. Several reports have been done at numerous locations to investigate the heavy metal content of the wastewater, and many of these reports mention that the heavy metal content exceeded the permissible limit. According to the majority of these reports, the type of heavy metal content varied depending on location, and one heavy metal may exceed standards while another does not. For example, wastewater in Saudi Arabia's Jazan region exceeded the limit for Cd and Mn [20], while the Fena River in Ghana's Ashanti region exceeded the limit for Cd, Pb, and Fe [21]. On the other hand, Huang *et al.* [22] reported that the Xiangjiang River, Hunan, China, had As, Cd, Sb, and Mn exceed the limit. As for the Miri River in Sarawak, Malaysia, it showed high contents of Fe, Mg, Cu, Mn, and Ni [23]. Although the remedy is not mentioned in some of the documented literature, we believe that the respective local authorities will take appropriate action and plan, as well as monitor the wastewater heavy metals issue. In addition, Table 3 shows the recent wastewater resource locations with heavy metals that exceeded the limit and the effects, along with remedies. The remedies include upgrading treatment plants, improving wastewater management and wastewater treatment facilities, continuous monitoring, developing new regulations, and so on [24–30].

2. Crosslinked polymer nanocomposites for heavy metal adsorption

2.1. Crosslinked polymer nanocomposites

Nanomaterials have unique properties, such as a large surface area with abundant functional groups and active sites that enable them to bond with various types of contaminants in the aqueous medium, making them good nano-adsorbents [31]. Although they show high capability for heavy metal adsorption,

Table 2. Heavy metal removal techniques [17, 18].

Method	Description	Advantages	Disadvantages
Chemical precipitation	Uses chemicals to react with heavy metals to form insoluble precipitates and remove them using sedimentation.	– Simple – Low cost – Effective in high concentration of heavy metal	– Produce sludge – Ineffective on low concentration of heavy metal – Ineffective on high pH value
Flotation	Air is introduced to wastewater to form micro-bubbles that attach the metal ions, inducing lower density agglomerates, raising the flocs to the top surface, and then they are removed.	– Selective metal ion recovery – Low sludge – High efficiency	– Inefficient on low concentration of heavy metal – Required additional chemical surfactant
Ion exchange	The heavy metal ion is removed by binding it to an immovable solid particle and replacing the solid particle's cation.	– Low sludge – Different resins can be used – No pH change	– High cost – Resin fouling
Coagulation-flocculation	Uses coagulants to destabilise and flocculants to agglomerate the destabilised particles, then remove them via sedimentation.	– High efficiency – Coagulating agent easily accessible	– High cost – Large consumption of chemical
Membrane filtration	It involves a membrane with a predetermined pore size and a driven force depending on the type of filtration process.	– High separation efficiency – No phase changes – Eco-friendly	– Low flow rates – High separation cost – Membrane fouling
Adsorption	Uses adsorbent for heavy metal in wastewater to adsorb on the surface upon contact.	– Low cost – Low sludge – Eco-friendly – High efficiency – Regeneration of adsorbent	– Highly depends on operating condition (temperature, pH value, amount of adsorbent, metal ions initial concentration)

Table 3. The issue of heavy metals in various wastewater locations.

Wastewater resources	Heavy metals	Effects	Remedies	Reference
Sungai Bunus, Kuala Lumpur, Malaysia	Cd, Cr, Fe, Mn	Carcinogenic – health concern	Upgrading treatment plant	[24]
Sungai Sembilang. Selangor, Malaysia	Al, Mn	Health concern	Water management	[25]
Nairobi, Kenya	Hg, Pb	Community health concern	Improving wastewater treatment infrastructure	[26]
Karnaphuli River, Chittagong, Bangladesh	Fe, Mn, Pb, Cd, Ni	Human health risk	Advice to improvise water management at the policy implementation level and conduct monitoring	[27]
River Yautepec, Mexico	Fe, Pb	Possible health risk	Continuous monitoring	[28]
Mgoua River, Douala, Cameroon	Cu, Cd, Pb	Negatively affected riverine ecosystem	Continuous monitoring on point sources and further treatment to be done before disposal.	[29]
Sewage sludge, Silesian Voivodeship, Poland	Zn, Cd, Ni, and Hg	Ecological risk	Develop new regulations and improvise the technological line of the wastewater treatment plant	[30]

some of the limitations of nano-adsorbents should be mentioned. For example, the nano-adsorbent itself has poor removal capacity as it is unstable and tends to aggregate. Hence, this leads to incorporating nanomaterials in different matrices to form a nanocomposite [32]. Polymer matrices exhibit numerous unique properties, such as tailorable porous structures, tuneable mechanical properties, and chemically bonded functional groups that provide special support for nanomaterials. Thus, the use of polymer nanocomposites is considered an effective and advanced method used for water treatment, particularly for heavy metal removal, as it possesses the advantages of both polymeric matrix and nanoparticles. In addition, the disadvantages of using solely free nanoparticles, such as dissolution, can be omitted [33].

Polymer nanocomposite is a mixture of more than two-phase materials in which the major phase is polymer, the dispersed phase is material of nanoscale size, and the performance of polymer nanocomposites is highly dependent on the state of distribution and dispersion of the nanoparticles in the polymer phase [34]. Further down into polymer nanocomposites, cross-linked polymer nanocomposites are nanocomposites with polymer matrices that undergo the cross-linking process with a crosslinking agent [35]. They possess excellent physical, chemical, mechanical, and electrical properties as compared to the typical ones. As such, they are highly utilized in various applications, including biomedical [36], construction [37], electronics [38], and automotive [39]. This review will focus on the usage of CPN in heavy metal removal from wastewater. With respect to their cross-linking nature, they possess a high free volume, do not dissolve in water, readily absorb water, and swell. The presence of functional groups along the network chains enabled them to have a high adsorption capacity for binding with heavy metal ions. These allow them to have a high potential for the application of heavy metal removal [40]. There are many possible routes in the preparation of crosslinked polymer nanocomposites. The preparation usually involves polymers, nanoparticles, and crosslinking agents.

The crosslinking of polymers can change the surface morphology and porosity and thus influence the final properties, *e.g.*, adsorption capacity. In some of the research findings, it was found that the modified polymeric materials (*e.g.*, chitosan) possess higher surface porosity upon modification using a crosslinking approach. Chitosan with a crosslinking agent added at a 25% concentration had a higher Cr (IV) adsorption capacity of 8.9 mg/g than that of uncrosslinked chitosan, with an adsorption capacity of 7.4 mg/g [41]. Although in the work of [41], there is no nanofiller added to the polymeric materials (*e.g.*, chitosan), it gives us a hint that crosslinking network formation (which may be controlled by the types and amount of crosslinking agent) can be used to increase the heavy metal adsorption capacity. Thus, it is worth furthering the research and development of this kind of crosslinked polymeric material with the addition of nanofiller, which could give us a deeper insight and potential evolution into CPN. Moreover, the report by Feng and Wen [42] also mentioned that the cross-linking density contributes to the overall performance of an adsorbent. In the report, starch xanthate was cross-linked with

N,*N′*-methylenebisacrylamide, and the adsorption performance of Pb^{2+} and Cd^{2+} was investigated with varied cross-linker contents. The adsorption for Pb^{2+} increased from approximately 44 mg/g to a maximum of 46.95 mg/g as the cross-linker content increased from 5 to 12 mg, then started to decline as the cross-linker content continued to increase. Similarly, Cd^{2+} reached a maximum of 36.05 mg/g from approximately 33 mg/g with 13 mg of crosslinker, then started to decline with crosslinker content. The reason proposed was that as the crosslinker reaches its optimum concentration, further increases in concentration will cause the saturation of crosslinking points to shrink and pores in the network to shrink, reducing adsorption capability.

Hyper-crosslinked polymers are porous, crosslinked polymeric materials that exhibit exceptional properties. Its micropore volumes and high surface area are achieved by extending the crosslinking further, resulting in a highly rigid network [43]. The dynamically crosslinked polymer has the distinctive structure of having dynamically reversible covalently bonded crosslinks. As a result, this kind of polymeric structure possesses unique properties such as self-healing and the ability for topological rearrangement by reshuffling the network joints while maintaining the 3-dimensional structure [44]. A hydrogel is a type of moderately cross-linked polymer containing abundant functional groups in the cross-linked network. These functional groups can achieve high-capacity adsorption of metal ions via hydrogen bonds, electrostatic force, and chemical complex interactions throughout the network [45]. Various types of cross-linked polymer nanocomposites can be developed based on an understanding of the structure-properties relationship of the polymer, the potential functionalization of the nanofiller (organic or inorganic), and the interaction between the polymer and nanofiller. Carboxylate, amine, carboxylic acid, hydroxyl, and even carbonyl groups can be used as functional groups. The heavy metal and the adsorbent will form a complex through electrostatic and ionic interactions [46].

The microstructure and surface morphology of CPN are varied depending on the types of polymeric materials, nanofiller, synthesis, and preparation methods, as well as the ways of microstructure examination (*e.g.*, microscopy with or without staining, cryo-fractured, *etc.*). Alkayal [47] investigated the surface morphologies of crosslinked polymethyl methacrylate/tin(IV) oxide (PMMA/SnO_2) using scanning electron microscopy (SEM). As shown in Figure 2, the microstructure of cross-linked-PMMA (C-PMMA) changed by increasing the loading of the

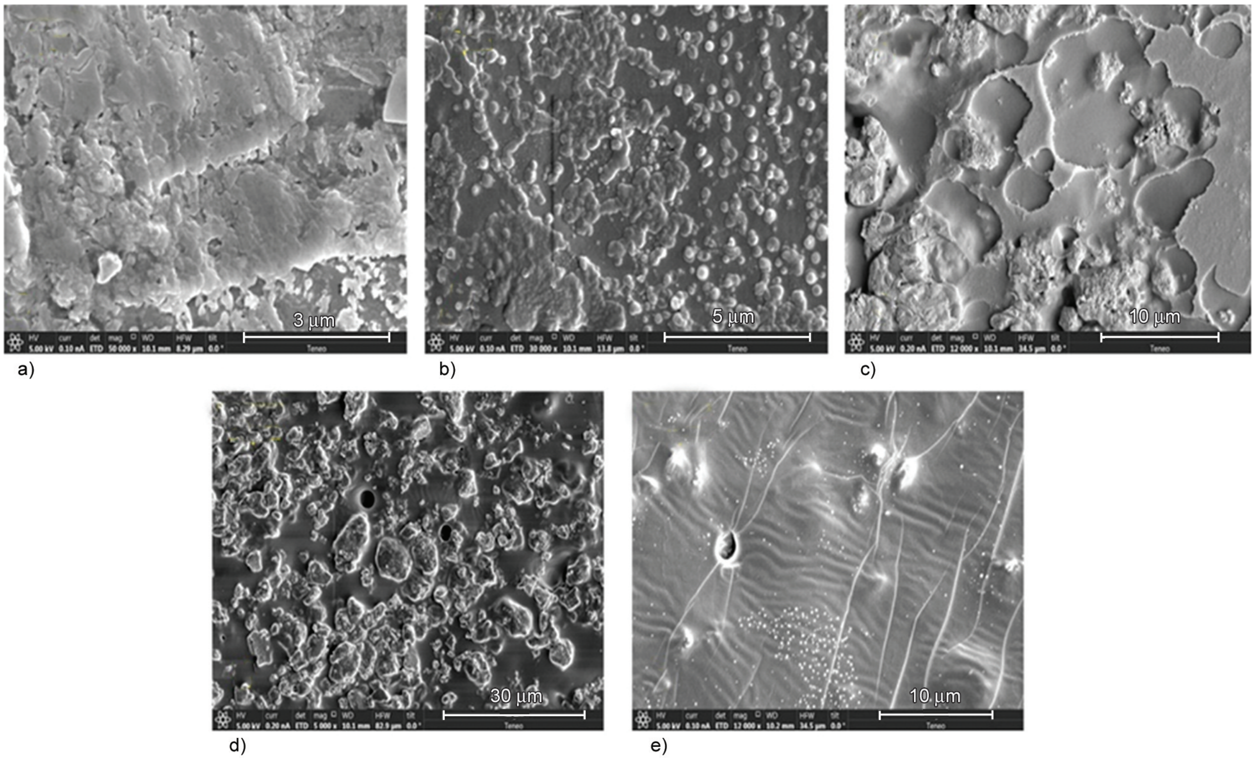

Figure 2. SEM images for a) C-PMMA (50 000×), b) C-PMMA/SnO_{2a} (30 000×), c) C-PMMA/SnO_{2b} (12 000×), d) C-PMMA/SnO_{2c} (5000×), and e) C-PMMA/SnO_{2d} (12,000×) [47].

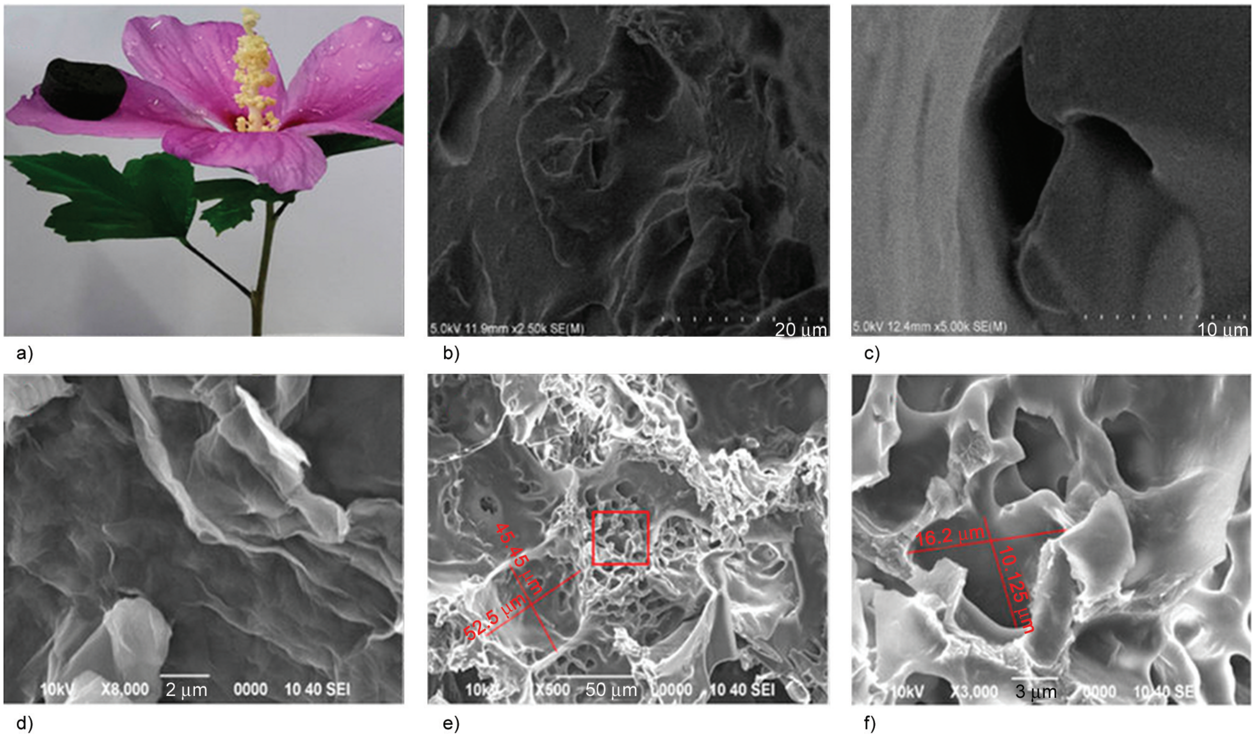

Figure 3. a) Photography of CLGO/PAA aerogel on the petal of the flower, the SEM image of: b), c), e) and f) of the CLGO/PAA aerogel in different magnifications, d) of the GO [48].

SnO_2 nanofiller (5, 10, 20, 40 wt%). The evolution of the microstructure was attributed to the interaction between the C-PMMA and SnO_2. Wang *et al.* [48] demonstrated the cross-section microstructure of the crosslinked graphene oxide/polyacrylic acid complex (CLGO/ PAA) using SEM. They found that the crosslinked materials possess a porous structure (c.f. Figures 3b and 3c). Figures 3e and 3f show that the CLGO/PAA has an interconnected, porous network, which can give a higher surface area and promote adsorption activity.

2.2. Wastewater heavy metal adsorption

Among all the available wastewater treatment methods for heavy metal removal, adsorption is one of the preferred methods due to its efficiency and cost-effectiveness. In terms of the heavy metal removal process, this method involves a mass transfer process by which the heavy metal is transferred from the liquid medium to the surface of the adsorbent and adsorbed via a physical or chemical attraction [49]. In terms of physical interaction, a weak physical force, such as the van der Waals force, randomly adsorbs on the surface of an adsorbent. On the other hand, chemical interactions involve different covalent or electrostatic bonds that attach the adsorbates to the surface of adsorbents [50]. Figure 4 illustrates a typical adsorption mechanism involving positively charged heavy metals adsorbed on negatively charged functional groups on the surface of an adsorbate via an electrostatic bond. At the same time, the adsorption process has an additional feature, which is the desorption process. It is a reverse process of adsorption whereby the adsorbate that is adsorbed on the adsorbent is removed, allowing the adsorbent to be reused once again. The desorption process, though, faces some difficulty as compared to the adsorption process, as the adsorbate has a high affinity towards the surface of the adsorbent as a result of the adsorption process [51]. From an economical and practical standpoint, the selection of the adsorbent significantly affects the efficiency and feasibility of both the adsorption and desorption processes. The adsorption mechanism of heavy metal adsorption via electrostatic and ionic interaction on the functional group surface

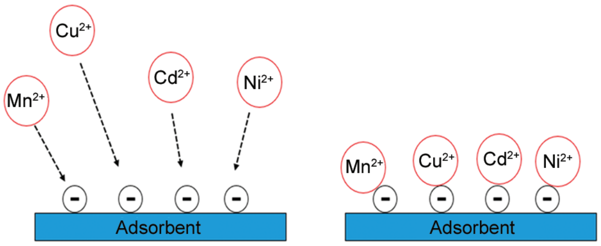

Figure 4. Adsorption mechanism via electrostatic bond.

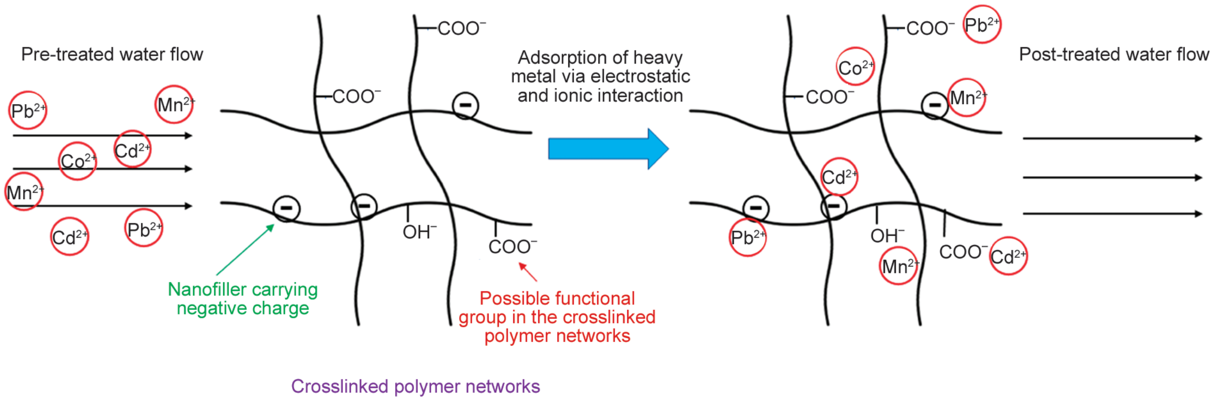

Figure 5. Possible mechanism of heavy metal adsorption on CPN.

of the crosslinking point and nanoparticles of a CPN is shown in Figure 5.

Table 4 summarises the research on several combinations of different crosslinked polymers and nanofillers. From Table 4, it can be seen that various types of polymer-nanofiller can be combined and crosslinked to achieve good heavy metal adsorption. Polymer (*e.g.*, polyethyleneimine, cellulose, sodium alginate, polyvinyl alcohol-*co*-polyacrylic) and nanofiller (*e.g.*, iron oxide nanoparticles, montmorillonite nanoclay) selection is an important criterion for increasing high adsorption capacity. Besides, from Table 4, it can be observed that each type of heavy metal has a different adsorption capacity for the same CPN. Thus, Table 4 can be used as a reference for CPN selection toward the removal of certain specific heavy metals. According to Mohsen *et al.* [52], polyvinyl alcohol-*co*-polyacrylic/montmorillonite irradiated with a ^{60}Co-source can achieve a Cu^{2+} adsorption capacity of up to 835 mg/g. The radiation was introduced to initiate the crosslinking reaction between the polymer matrices. As the dose of radiation increases, the density of crosslinks increases due to the formation of more free radical sites.

The water swelling capability of the CPN was investigated in relation to its adsorption capacity. The swelling percentage increases with the crosslink density to a certain extent and then decreases. The increase is associated with the ease of solvent diffusion, but to a certain extent, the swelling process was restricted due to the higher crosslink density. This suggests that crosslink density facilitates the swelling process to a certain extent before the restriction of swelling starts to occur. It should be noted that comparing research works may be difficult due to the various parameters used during their work, such as different heavy metals, pH (*e.g.*, pH 5, pH 6), and testing duration (*e.g.*, 30 minutes, 2 hours, 24 hours, and so on). Nevertheless, all this literature can be used as a clue to understanding the behavior of the CPN in heavy metal adsorption.

Zhang and Li [53] prepared poly(trans-aconitic acid/2-hydroxyethyl acrylate)/nanosized hydrous ferric oxide and discovered that Pb^{2+} and Cu^{2+} adsorption could be as high as 303.76 and 107.52 mg/g, respectively. It was suggested that the micropores in the matrix with 3D network microstructures and the abundance of hydroxyl groups in the hydrous ferric oxide favor the adsorption process. Similarly, Li *et al.* [54] fabricated an interpenetrating polymer network hydrogel to remove Pb^{2+} and Cu^{2+}. The hydrogel was synthesized by crosslinking poly(2-(dimethylamino)-ethyl methacrylate and nanofibrillated cellulose. The findings showed that a higher loading of the nanofibrillated cellulose not only introduced more carboxyl groups but also increased the porosity and surface area of the CPN that facilitated the adsorption. On the other hand Chai *et al.* [55] investigated the removal of arsenic ions using a CPN that consists of 2,2,6,6-tetramethylpiperidine-1-oxyl (TEMPO) treated nanocellulose (TEMPO-NC) crosslinked with glutraldehye (GA) and branch polyethyleneimine (PEI). The Langmuir model showed that the adsorption capacity could reach up to 255.19 mg/g. This CPN was able to perform differently than other CPNs as the adsorption increased with the reduction of pH. In their study, it was found that the crosslinked system at pH 3 showed the highest adsorption as compared to other pHs. It was suggested that it was due to its unique structure that, at low pH, NH_2 around the structure generates an intense repulsive force with neighboring polymer molecules. As a result, the space between polymer molecules increased, which helped to increase the cavity for diffusion and adsorption capacity instead.

In the work by Kabir *et al*. [56], tetraethylenepentamine methacrylamide and nanocrystalline cellulose were cross-linked with *N*,*N′*-methylenebisacrylamide. The adsorption capacity was 26.5 mg/g at pH 3.8 and 52.5 mg/g at pH 10. This may be attributed to the presence of different kinds of functional groups (such as hydroxyl, carboxyl, and amine) on the surface of the CPN that contributed to the heavy metal ion adsorption capacities. Besides, work on the removal of Cu^{2+} ions using CPN was done by Mo *et al*. [57]. The CPN was synthesized by self-assembly of oxidized nanofibrils crosslinked with trimethylolpropane-tris-(2-methyl-1-aziridine) propionate and polyethylenimine (PEI). For Cu^{2+} ions, the adsorption capacity was as high as 485.44 mg/g. This is mainly due to the 3D multi-wall porous structure, which allows the diffusion of Cu^{2+} from the outside into the inner site. In addition, the abundance of amino and oxygen-containing functional groups on the surface of the aerogel helps to bind the Cu^{2+} ions onto the surface. Mahmoud *et al*. [58] created a CPN from iron oxide waste nanoparticles and alginate. The adsorption experiment was also performed on different water sources, such as tap water, seawater, and industrial water. The outcome showed

Table 4. Crosslinked polymer nanocomposites (CPN) and their capacity for heavy metal adsorption (the concentration marked as C_0).

Heavy metal	CPN	Preparation	Adsorption capacity	References
Pb^{2+}	Sodium alginate/iron oxide waste nanoparticles	Solution mixing with formaldehyde as crosslinking agent at 25 °C and dried overnight Sample form: fine powder	564 mg/g Experimental condition: – using 20 mg nanocomposite – C_0 = 41.44 g/l (converted from C_0 = 0.2 mol/l) – pH = 6 – time = 30 minutes	[58]
	Poly(trans-aconitic acid/2-hydroxyethyl acrylate)/nanosized hydrous ferric oxide	– Crosslink via ^{60}Co-γ source – Hydrogel mixed and stirred with nanofiller in a thermostatic oscillator at 25 °C for 24 h (150 rpm) Sample form: hard gel	303.76 mg/g (converted from 1.466 mmol/g) Experimental condition: – using 0.05 g nanocomposite – C_0 = 0.829 g/l (converted from C_0 = 4 mmol/l) – Temperature = 25 °C – Time = 24 h	[53]
	Nanocellulose/poly(2-(dimethylamino)ethyl methacrylate	Crosslinking free radical polymerization of poly(2-(dimethylamino)ethyl methacrylate) in nanofibrillated cellulose suspension Sample form: hydrogel	81.96 mg/g Experimental condition: – using 0.02 g nanocomposite – pH = 5 – Temperature = 25 °C – Time = 48 h	[54]
Cd^{2+}	Sodium alginate/iron oxide waste nanoparticles	Solution mixing with formaldehyde as crosslinking agent at 25 °C and dried overnight Sample form: fine powder	102.2 mg/g Experimental condition: – using 20 mg nanocomposite – C_0 = 22.48 g/l (converted from C_0 = 0.2 mol/l) – pH = 5 – Time = 30 minutes	[58]
	Poly(trans-aconitic acid/2-hydroxyethyl acrylate)/nanosized hydrous ferric oxide	– Crosslink via ^{60}Co-γ source – Hydrogel mixed and stirred with nanofiller in a thermostatic oscillator at 25 °C for 24 h (150 rpm) Sample form: hard gel	150 mg/g (converted from 1.333 mmol/g) Experimental condition: – using 0.05 g nanocomposite – C_0 = 0.450 g/l (converted from C_0 = 4 mmol/l) – Temperature = 25 °C – Time = 24 h	[53]

Table 4. Continuously 1

Heavy metal	CPN	Preparation	Adsorption capacity	Reference
Cu^{2+}	Sodium alginate/iron oxide waste nanoparticles	Solution mixing with formaldehyde as crosslinking agent at 25 °C and dried overnight Sample form: fine powder	158 mg/g Experimental condition: – using 20 mg nanocomposite – C_0 = 22.71 g/l (converted from C_0 = 0.2 mol/l) – pH = 6 – Time = 30 minutes	[58]
	Poly(trans-aconitic acid/2-hydroxyethyl acrylate)/nanosized hydrous ferric oxide	– Crosslink via ^{60}Co-γ source – Hydrogel mixed and stirred with nanofiller in a thermostatic oscillator at 25 °C for 24 h (150 rpm) Sample form: hard gel	108 mg/g (converted from 1.692 mmol/g) Experimental condition: – using 0.05 g nanocomposite – C_0 = 0.254 g/l (converted from C_0 = 4 mmol/l) – Temperature = 25 °C – Time = 24 h	[53]
	Organic montmorillonite-polyvinyl alcohol-*co*-polyacrylic	Solution mixing and irradiated with ^{60}Co-γ source Sample form: hydrogel films	835 mg/g Experimental condition: – using 0.5 g nanocomposite – C_0 = 0.1 g/l (converted from C_0 = 100 ppm) – pH = 5 – Time = 2 h	[52]
	Nanocellulose/poly(2-(dimethylamino)ethyl methacrylate	Crosslinking free radical polymerization of poly(2-(dimethylamino)ethyl methacrylate) in nanofibrillated cellulose suspension Sample form: hydrogel	217.39 mg/g Experimental condition: – using 0.02 g nanocomposite – pH = 5 – Temperature = 25 °C – Time = 48 h	[54]
	Oxidized cellulose nanofibril/crosslinked trimethylolpropane-tris-(2-methyl-1-aziridine) propionate/polyethyleneimine (TO-CNF/TMPTAP/PEI)	The TO-CNF reacted with TMPTAP via a ring-opening reaction in an aqueous solution at room temperature and then cross-linked with PEI at 30 °C Sample form: aerogel	485.44 mg/g Experimental condition: – using 0.2 g/l nanocomposite – C_0 = 0.5 g/l – pH = 5.5 – Temperatuare = 25 °C – Time = 24 h	[57]
Ni^{2+}	Poly(trans-Aconitic acid/2-hydroxyethyl acrylate)/nanosized hydrous ferric oxide	– Crosslink via ^{60}Co-γ source – Hydrogel mixed and stirred with nanofiller in a thermostatic oscillator at 25 °C for 24 h (150 rpm) Sample form: hard gel	85.87 mg/g (converted from 1.463 mmol/g) Experimental condition: – using 0.05 g nanocomposite – C_0 = 0.235 g/l (converted from C_0 = 4 mmol/l) – Temperature = 25 °C – Time = 24 h	[53]
	Organic montmorillonite-polyvinyl alcohol-*co*-polyacrylic	Solution mixing and irradiated with ^{60}Co-γ source Sample form: hydrogel films	636 mg/g Experimental condition: – using 0.5 g nanocomposite – C_0 = 0.1 g/l (converted from C_0 = 100 ppm) – pH = 5 – Time = 2 h	[52]
Fe^{2+}	Tetraethylenepentamine methacrylamide/nanocrystalline cellulose	Solution mixing Sample form: hard gel	26.5 mg/g Experimental condition: – using 4 g/l nanocomposite – C_0 = 450 mg/l – pH = 3.8	[56]

Table 4. Continuously 2

Heavy metal	CPN	Preparation	Adsorption capacity	Reference
As^{5+}	Nanocellulose-branched polyethyleneimine/ glutaraldehydes	Solution mixing with glutaraldehydes as crosslinking agent 25 °C for 0.5 h Sample form: solid particles	255.19 mg/g Experimental condition: – using 20 mg nanocomposite – C_0 = 0.3 g/l – pH = 3 – Temperature = 25°C – Time = 6 h	[55]
Co^{2+}	Organic montmorillonite-polyvinyl alcohol-*co*-polyacrylic	Solution mixing and irradiated with ^{60}Co-γ source. Sample form: hydrogel films	785 mg/g Experimental condition: – using 0.5 g nanocomposite – C_0 = 0.1 g/l (converted from C_0 = 100 ppm) – pH = 5 – Time = 2 h	[52]

that the CPN's performance is stable despite having trace amounts of different elements in varying sources of water.

2.3. Factors influencing CPN's performance for heavy metal adsorption

2.3.1. Functional groups and structures of polymer matrices

The performance of cross-linked polymer nanocomposites is determined by adsorbent properties such as the polymer matrices and fillers used. In the selection of polymers in a composite, the type of functional group and the number of active sites, along with the chemical structure of the polymer, heavily affect the efficiency of heavy metal adsorption. In terms of functional groups in polymers, carboxylic acids, amines, hydroxyls, and sulphonic acids are extremely crucial in the efficiency of heavy metal adsorption, thus making the selection of polymers important in CPN. According to the hard-soft acid-base theory, heavy metals can be classified as soft or hard acids based on whether they prefer to bind with their respective bases. For example, chitosan is rich in amine and hydroxyl groups that are capable of heavy metal adsorption. In addition, it can be easily crosslinked with a crosslinking agent, which can decrease its solubility towards most organic and mineral acids [59]. The structure of the polymer adsorbent has significant effects on the adsorption process as well.

Cahyaningrum *et al.* [60] compared the adsorption capacities of chitosan in the form of beads and powder towards Mg^{2+}. The adsorption capacities of chitosan in the form of beads and powder are 42.26 and 17.50 mg/g, respectively. The difference in this was related to the expansion of surface area through swelling and the increased number and accessibility of active sites. In the work done by Hassan *et al.* [61], they incorporated halloysite nanotube and ball-milled biochar into alginate and crosslinked it using calcium ions, which successfully synthesized a mesoporous crosslinked nanocomposite with maximum adsorption capacities of Pb, Cu, Cd, and Ni of approximately 26.49, 16.87, 6.96, and 2.85 mg/g, respectively. The Langmuir surface area was shown to decrease from pre-adsorption as compared to post-adsorption, from 38.71 to 15.71 m^2/g, proving the significance of the mesoporous structure in contributing to the adsorption process.

2.3.2. Effects of nanofillers

Yu *et al.* [62] investigated the effects of graphene oxide (GO) on the crosslinked polyvinyl alcohol (PVA) containing GO and sodium alginate (SA). PVA/SA hydrogel recorded 230.9 mg/g of Pb^{2+} adsorption, while the adsorption capacity increased with the increased amount of GO. The adsorption capacity of the crosslinked PVA increased up to 279.43 mg/g in the presence of 5 wt% GO. This is associated with the strong complexation between Pb^{2+} and –COO on the surface of GO-SA, as well as the high surface area and a loose structure of the crosslinked polymer system.

Besides, different types of nano-adsorbents, such as carbon-based nanomaterials, silica-based nanomaterials, zero-valent metal-based nanomaterials, and metal oxide-based nanomaterials, are great nano-adsorbents for heavy metals, and each of them possesses its own unique properties that contribute to the heavy metal removal process [63]. Carbon-based nanomaterials such as carbon nanotubes (CNT) are known as excellent nano-adsorbents due to their unique properties such as large surfaces, high porosity with a hollow structure, and the ability to develop a strong interaction in the adsorption of heavy metals. In the research done on single-wall carbon nanotubes (SWCNT), Moradi [64] found that the adsorption capacities of Pb^{2+}, Cu^{2+}, and Cd^{2+} were 33.55, 24.29, and 24.07 mg/g, respectively, through the physisorption process in relation to the hollow structure of SWCNT. The difference in heavy metal adsorption capacity was also explained in terms of cation size, with the size increase in the order of Pb^{2+}, $Cu2^{+}$, and Cd^{2+}, resulting in higher adsorption on Pb^{2+}. The adsorption capacity between different nanofillers differs as it heavily depends on the size, structure, surface area, and functional group of the nanofiller itself. Nevertheless, as mentioned previously, the adsorption of certain types of heavy metals is also governed by the hard-soft-acid-base theory.

Thus, the performance of nanofillers in heavy metal adsorption can be further improved by introducing more active sites and different functional groups through surface modification or functionalization. The adsorption of heavy metals is particularly effective on functional groups such as amine, amide, carboxyl, and sulfonic. Thus, introducing such functional groups becomes another approach to improve the efficiency of heavy metal adsorption. To investigate this effect, Lin *et al*. [65] conducted research based on corncob and corncob grafted with polyacrylamide. The adsorption capacity of cadmium increased from 4.96 to 18.35 mg/g after the modification as a result of the increment in the functional group and adsorption site. In the research by Xie *et al*. [66], carbon nanotubes were coated with polydopamine and surface-modified with an amino group from polyethylenimine. An abundance of hydrophilic functional groups on the CNT surface can promote its dispersibility in water and adsorption capability. From the results obtained, the functionalized CNT had higher adsorption toward Cu^{2+} compared to the unmodified CNT, with an adsorption capacity of 34.6 and 25.4 mg/g, respectively. Likewise, researchers also did functionalization on the crosslinked polymer as well. In the research by Abadast *et al*. [67], the introduction of the thiol grafted group in the 3D-network polymer showed an improvement in the adsorption of Hg^{2+} compared with the 3D-network polymer without functionalization, with an approximate maximum removal percentage of 98 and 40%, respectively.

2.3.3. Adsorption time

Besides the adsorbent properties, the conditions of the adsorption process also significantly affect the adsorption capacity. Adsorption is a time-dependent process in which contact time influences the adsorption process. At the beginning of the process, there will be a massive number of sorption sites on the adsorbent surface, and they will promote rapid external diffusion. However, as time goes on, the available sorption sites will decrease, causing a decline in the adsorption process until equilibrium is achieved [68].

2.3.4. Initial pH value

Furthermore, the metal adsorption effect is dependent on the initial pH value of the aqueous medium. In the current scenario, industrial wastewater is not always at a pH value of 7. Thus, the variation in pH value may influence the adsorption performance. Jiang *et al*. [69] reported that the adsorption capacity of glucan/chitosan hydrogel on copper, cobalt, nickel, lead, and cadmium ions increases as the pH value increases, and the increment is larger at a higher pH value compared to a lower pH value. This is mainly due to the chemical speciation of the metal ions in the medium. At a lower pH value of the aqueous medium, there will be a higher concentration of H^{+} ions, which compete with the available metal ions at the adsorption site. Besides, a lower pH value also causes the ionization of certain functional groups to become protonated around the surface of the adsorbent, restricting the adsorption of other metal ions [70]. As such, pH correction is often used in wastewater treatment plants before it is conveyed for further treatments that are dependent on the pH value. This is done by thoroughly mixing the wastewater and neutralizing chemicals such as sulfuric acid, hydrochloric acid, sodium hydroxide, or ammonium hydroxide, depending on the pH value intended [71].

2.3.5. Temperature

Temperature plays a role in several processes in wastewater treatment, especially the rate of the conversion process and the efficiency of adsorption and removal. Although the temperature factor is not as significant as other aspects of wastewater treatment, like biological treatment, the temperature of the metal-ion aqueous medium influences the adsorption capacity of a particular adsorbent. For an endothermic adsorption process, the increase in temperature will promote the higher mobility of the heavy metal ions in the adsorption process, leading to a higher adsorption capacity. On the other hand, the exothermic process will have a decrease in adsorption capacity as the temperature increases [72]. Even so, the actual temperature of wastewater is not always constant and may vary with climate and location. An actual case study was done by Shahat and Al-Najar [73] regarding the impact of temperature variation in the Gaza Strip in the Mediterranean region. The average daily wastewater temperature ranges from 25 to 13 °C during the summer and winter, respectively.

In terms of CPN feasibility, Nasef *et al*. [74] investigated the temperature effect of crosslinked polyethylene-graft-polystyrene sulfonic acid in the range of 25 to 65 °C. From the result obtained, the crosslinked adsorbent showed a maximum adsorption capacity of 68, 65, 61, 55 g, and 49.0 mg/g towards Ni^{2+}, Co^{2+}, Cu^{2+}, Pb^{2+}, and Ag^{+}, respectively. The adsorption capacity showed tiny to insignificant changes from 25 to 65 °C, which suggested that the adsorption mainly took place by a physical process.

3. Challenges and future perspective of CPN

The adsorption method is widely used in wastewater heavy metal adsorption as it demonstrates some advantages over the other available methods. These include its wide range of design options and the availability of adsorbents in terms of polymer matrices and nanofillers to suit the intended purpose. The crosslinked structure of matrices and the addition of a high-surface-contact nanofiller have been proven to enhance the adsorption capacity of an adsorbent. Despite the great heavy metal adsorption capacity in CPN, the performance of CPN depends on the dispersion and distribution of nanofiller throughout the nanocomposite. Nanofillers have the tendency to agglomerate, which reduces the surface area and active sites for adsorption. For instance, most of the research performed on CPN adsorption capacity is done in a lab-based, designed environment. This includes the pre-determined conditions such as the pH value, temperature, *etc.* In an actual application, the conditions may vary and render different performances. Furthermore, the content of actual water resources contains a mixture of heavy metals and other impurities that may interfere with the adsorption process. Al Hamouz *et al*. [75] successfully synthesized a highly crosslinked bisphenol-S paraformaldehyde piperazine (BSPF) polymer, which was registered under a Justia US patent (publication number: 20190092892) as an effective heavy metal adsorbent. The adsorption capacity was tested in actual industrial wastewater from Dammam, Saudi Arabia, with various heavy metal contents in the wastewater that varied in initial concentration. The findings showed that Pb^{2+} achieved a percentage removal of ~99% with an initial concentration of ~2690 µg/l while other heavy metals ranged from ~45 to ~76% percentage removal at different initial concentrations. According to the finding, crosslinked polymers have a higher potential for heavy metal adsorption. Nevertheless, the adsorption of certain crosslinked polymers alone is rather selective for heavy metal adsorption, with average performance for other heavy metals. Based on the summary of previous works done by other researchers, the addition of nanofillers to a crosslinked polymer system is believed to improve the overall and wider range of heavy metal adsorption through the use of synergistic mechanisms.

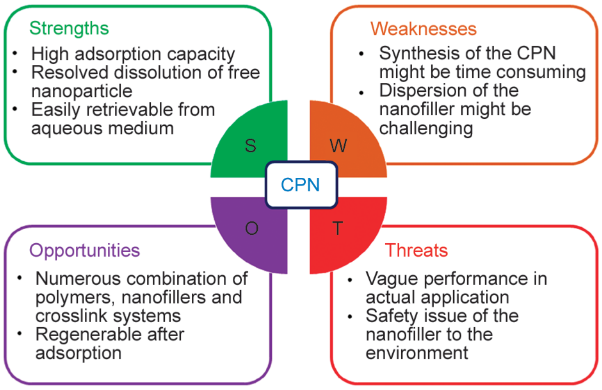

Figure 6. SWOT analysis of CPN.

To summarise this, Figure 6 illustrates the SWOT (strengths, weaknesses, opportunities, and threats) analysis of the CPN. Normally, SWOT analysis is used for strategy and planning for personal or institutional management. Here, we do the SWOT analysis for the CPN for us to understand more about the CPN. This would help us develop a better and higher-performing CPN in academic and industrial areas.

4. Conclusions

CPN emerged as a promising adsorbent in the heavy metal adsorption application after combining crosslinked polymers and nanofillers. It has great design flexibility. Compared with other uncrosslinked polymer nanocomposites, CPN exhibits better stability, both physically and chemically. CPN can also readily absorb water and swell with better adsorption capacity to a certain extent of crosslink density. The performance of numerous combinations in terms of different types of polymers and nanoparticles is worth exploring. Cost-effectiveness in the selection of materials should also be a deciding factor in the actual applications. In terms of adsorbent recyclability, the performance of the desorbing agent used in each CPN may differ, and thus, the selection of a suitable desorbing agent should also be considered in terms of its compatibility, cost-effectiveness, number of cycles, and environmental friendliness. In the future, it is recommended to have more adsorption tests in actual application conditions and explore different kinds of desorbing agents (*e.g.*, sodium nitrate, sodium acetate, nitric acid, and hydrochloric acid) to further increase the overall usage and efficiency of CPN, which could benefit the environment.

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