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## **Polyaniline-based adsorbents for aqueous pollutants removal: A review**

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### **Abstract**

Water contamination by a variety of pollutants, such as conventional pollutants (e.g. heavy metals and organics) and emerging micropollutants (e.g. personal care products and endocrine disrupting compounds) is of great concern. Various technologies have been developed to address this critical challenge. Among them, adsorption by polyaniline (PANI)-based nanocomposites, has gained growing research interest recently due to their unique electrical characteristics and many other benefits, such as facile synthesis, low cost, excellent environmental stability, simple acid-base doping/dedoping process, reactive NH- groups, and tunable properties. This paper provides a comprehensive review of the typical PANI-based adsorbents and their properties. Moreover, the removal of various aqueous contaminants (including heavy metals and metalloids, organic dyes, and emerging pollutants) by PANI-based adsorbents is discussed and assessed. The unique doping/dedoping of PANI-based adsorbents alters the morphology, pore size, surface charge, and functional groups of the adsorbents, leading to enhanced adsorbent-adsorbate interactions and thus adsorption performance. The adsorption mechanisms of aqueous pollutants by PANI-based adsorbents include electrostatic interactions, van der Waals forces,  $\pi$ - $\pi$  interactions, hydrogen bonding, chelating and/or

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complexation effects. The kinetic studies by pseudo-first order, pseudo-second order, Elovich, and intraparticle diffusion models and regeneration of PANI-based adsorbents are also discussed. Finally, the future outlooks on aqueous pollutants removal by PANI-based adsorbents are presented.

**Keywords:** polyaniline; water treatment; heavy metal removal; organic dyes; emerging pollutants.

## 1. Introduction

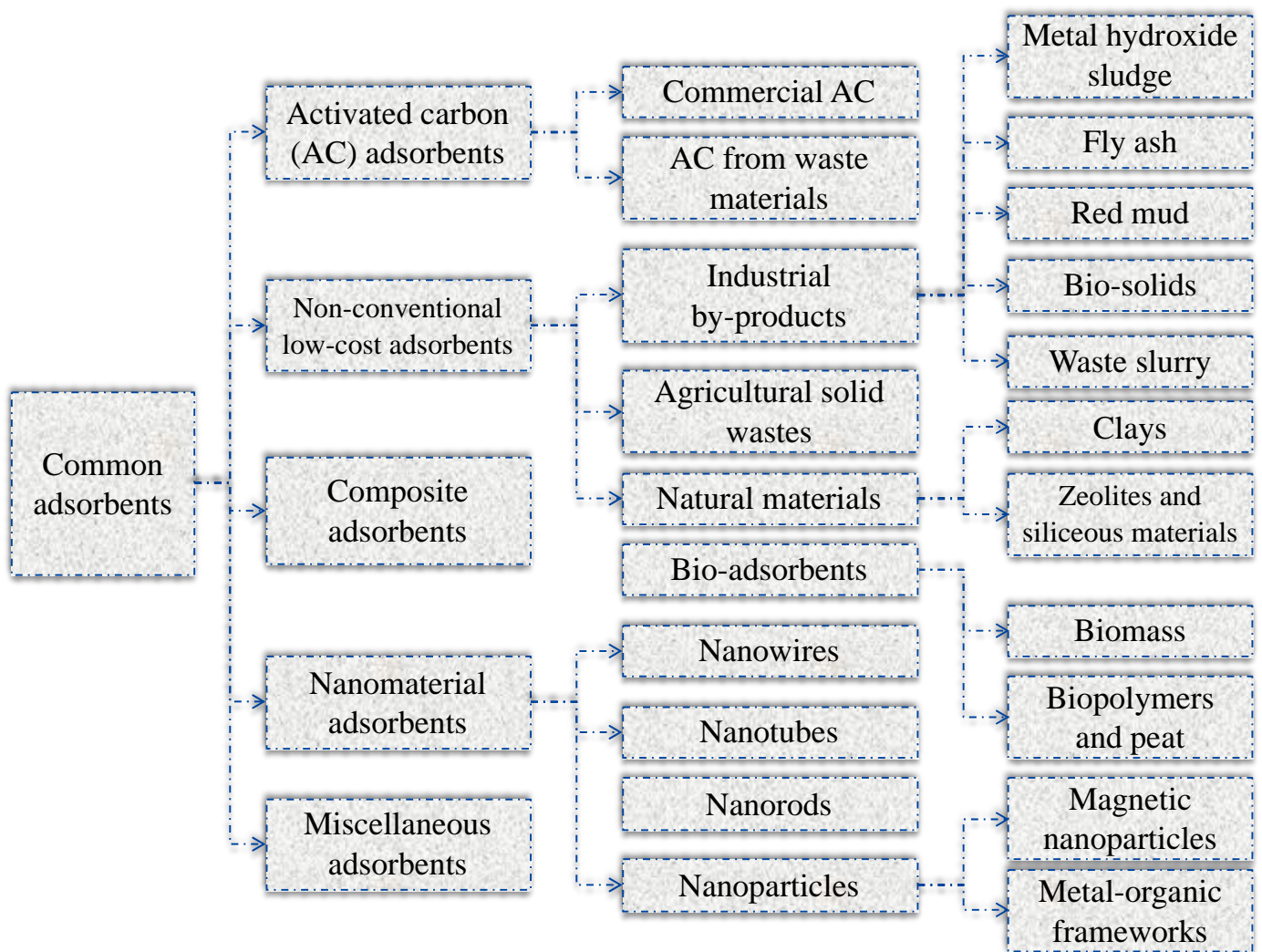
### *1.1. Water contamination and adsorption*

Freshwater is of vital importance for humans as it drives not only all lives but also all activities of these lives. Although more than 70% of the Earth's surface is covered by oceans and seas, only 0.8% of the Earth's water is considered as fresh water [1]. Nowadays over 1 billion people have little access to clean drinking water and approximately 2.3 billion people live in water-stressed regions [2]. Various organic and inorganic pollutants, such as heavy metals, dyes, humic substances, phenolic compounds, pesticides, herbicides, pharmaceuticals, per- and polyfluoroalkyl substances (PFASs), and polychlorinated biphenyls (PCBs) cause severe water contamination, which worsens water scarcity and shortages [3, 4].

A variety of technologies, such as adsorption, membrane filtration, photo-remediation, and ion exchange have been developed to remove pollutants from water [5, 6]. Adsorption is one of the most cost-effective and promising methods because of its low cost, insensitivity to toxic pollutants, simplicity, and flexibility in design and operation [7]. The past decades have witnessed emerging research activities to develop numerous materials for adsorption of aqueous pollutants [8]. The most common adsorbents for the removal of aqueous

pollutants are illustrated in Fig. 1. Various adsorbents, such as activated carbon, natural zeolites, alumina, biochar, and clay minerals have been used for the removal of aqueous pollutants. However, their applications are limited by some challenges, such as the low adsorption capacities, high costs and regeneration issues [7].

Thus, growing research interest has been shifted towards low-cost and efficient adsorbents with facile regeneration. Recently, conducting polymers, such as polyaniline (PANI) and polypyrrole (PPy) have attracted increasing attention for aqueous pollutants removal because of their simple synthesis procedures, tunable morphologies and versatile functional groups [8]. This review details and evaluates PANI and its composites due to their low costs, ease of synthesis, excellent environmental stabilities, and unique doping/dedoping properties [8].



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Fig. 1. Summary of common adsorbents for the removal of aqueous pollutants, modified from [9].

## ***1.2. Polyaniline (PANI)-based nanocomposites for water and wastewater treatment***

Polymers are a class of new materials for adsorption because of their high surface areas, adjustable surface chemistry, excellent mechanical rigidity, desirable pore size distribution, and economical regeneration under mild conditions [10]. Polyaniline (PANI), a conducting polymer, has been extensively studied as a promising adsorbent for aqueous pollutants removal due to its unique electrical characteristics, facile synthesis, low costs of monomers, excellent environmental stability, effortlessly-controlled reversible properties (e.g. by charge exchange doping and protonation), unique functional groups (e.g. NH-), and tunable properties [11-15]. In addition, PANI-based nanocomposites have also been widely studied for adsorption of pollutants, due to their high surface areas, excellent dispersibility, and synergistic properties of the polymer and the filler [8, 15, 16]. Another advantage of PANI-based nanocomposites is their diverse morphological structures, which are adjustable and predictable by key synthesis parameters, such as the dopant type, pH, contact time, temperature, adsorbent dosage, concentration, nature of the adsorbent, and the presence of competing ions. Consequently, PANI-based nanocomposites with various nanostructures have been fabricated for aqueous pollutants removal (e.g. heavy metals and dyes) [8, 11, 14].

Increasing interest in PANI-based nanocomposites for the removal of aqueous pollutants in the past two decades is illustrated in Fig. 2A. Fig. 2B shows the contributions from different countries/regions on the investigation of PANI-based adsorbents. A number of reviews have been published and covered different aspects of the treatment of aqueous pollutants, such as heavy metals [10, 16-21], dyes [10, 15, 17, 18], and emerging pollutants [22-24] by various nanostructured adsorbents, such as conductive polymers [5, 10], clay-based adsorbents [25], biodegradable polymers [10, 18, 26], graphene nanosheets [22], and carbon nanotubes [27]. However, few reviews focusing on PANI-based nano-adsorbents for aqueous pollutants removal have

been published so far.

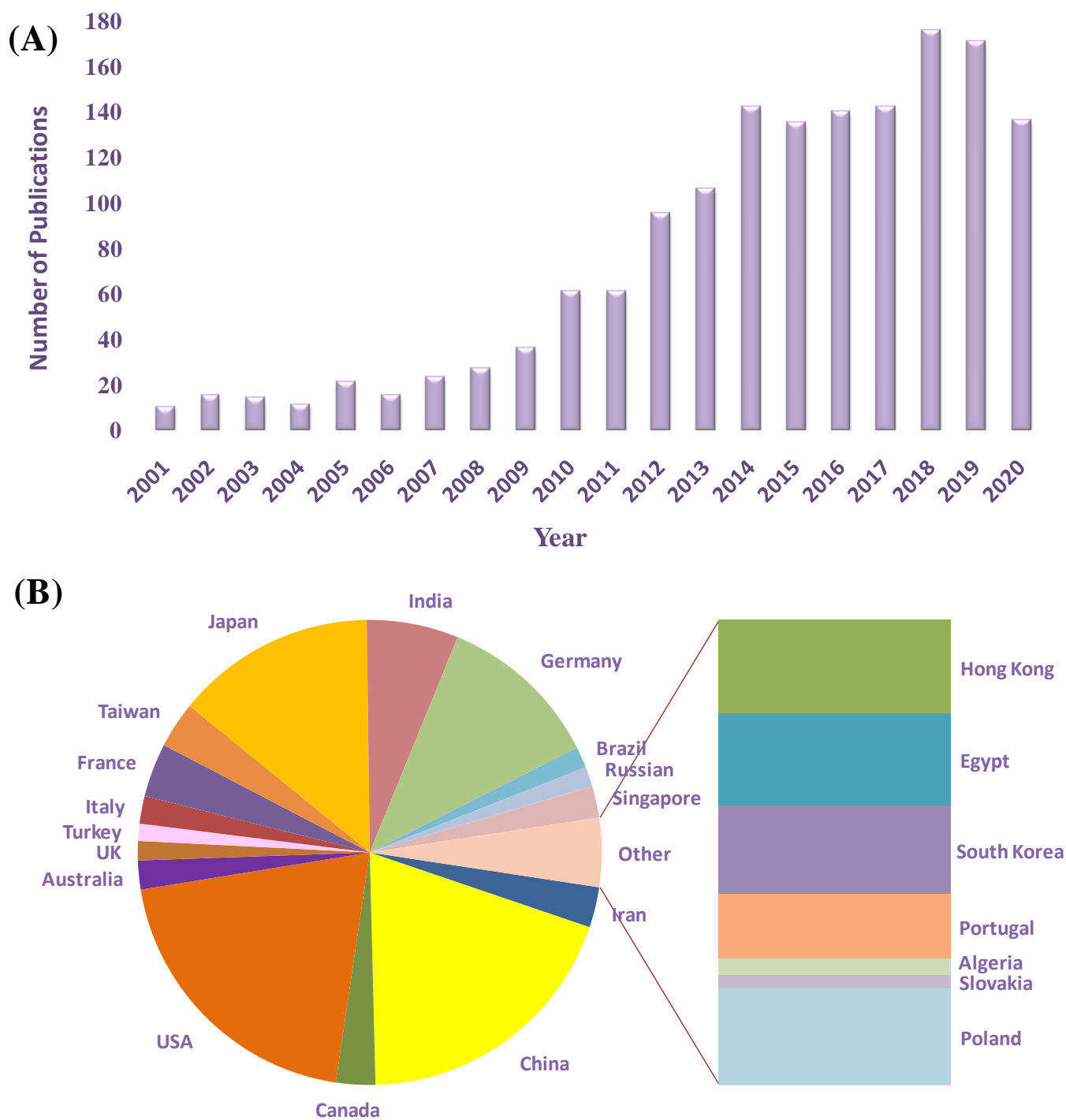


Fig. 2. (A) Growing research interest in PANI-based adsorbents for pollutants removal. The data are obtained from Google Scholar using “polyaniline” or “PANI” as the keyword for searching. (B) Contributions from different countries/regions on the investigation of PANI-based adsorbents.

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This comprehensive review focuses on the removal of aqueous pollutants from water and wastewater. Moreover, the variety of PANI polymer and its derivatives, PANI copolymers, and PANI-based nanocomposites with organic and inorganic materials are discussed for the treatment of a diversity of pollutants. The removal of various pollutants is discussed in three main categories of (I) heavy metals and metalloids, (II) organic dyes, and (III) emerging pollutants. Furthermore, the adsorption mechanisms and driving forces for the removal of pollutants are discussed for each type of adsorbent as well as for each category of the pollutants.

## **2. Types for polyaniline-based adsorbents**

### **2.1. Polyaniline (PANI)**

Various properties of PANI, such as mechanical, electrical, optical, physical, thermal, chemical, and morphological properties have been investigated in many applications [28-32]. For PANI-based adsorbents, the most studied properties are the surface area, surface charge, hydrophobicity, morphology, and functional groups. PANI, a representative from the family of conducting polymers, is distinguished by easy synthesis and high environmental stability [33]. The unique features of PANI are related to its highly ordered chemical structure. Chemically, PANI consists of “ $n$ ” reduced benzenoid diamine and “ $m$ ” oxidized quinoid diamine repeating units, where the oxidation state of PANI depends on the value of “ $m$ ”. PANI exists in three oxidation states: leucoemeraldine, pernigraniline, and emeraldine having  $m:n$  ratio as 0:1, 1:1, and 1:0, respectively with different conductivities and electric charges (Fig. 3A) [13-15, 34, 35]. Predominately imine groups but also the amine groups in PANI chains can be further protonated in the presence of  $H^+$  (acidic) ion or the dopant to generate the cationic defects that are the main reason for the reversible conductivity of PANI [34]. The unprotonated and protonated forms of PANI are known as base and salt, respectively (Fig. 3B). Emeraldine is

the most stable form of PANI, containing an equal number of the oxidized and reduced units [11, 13, 15, 35].

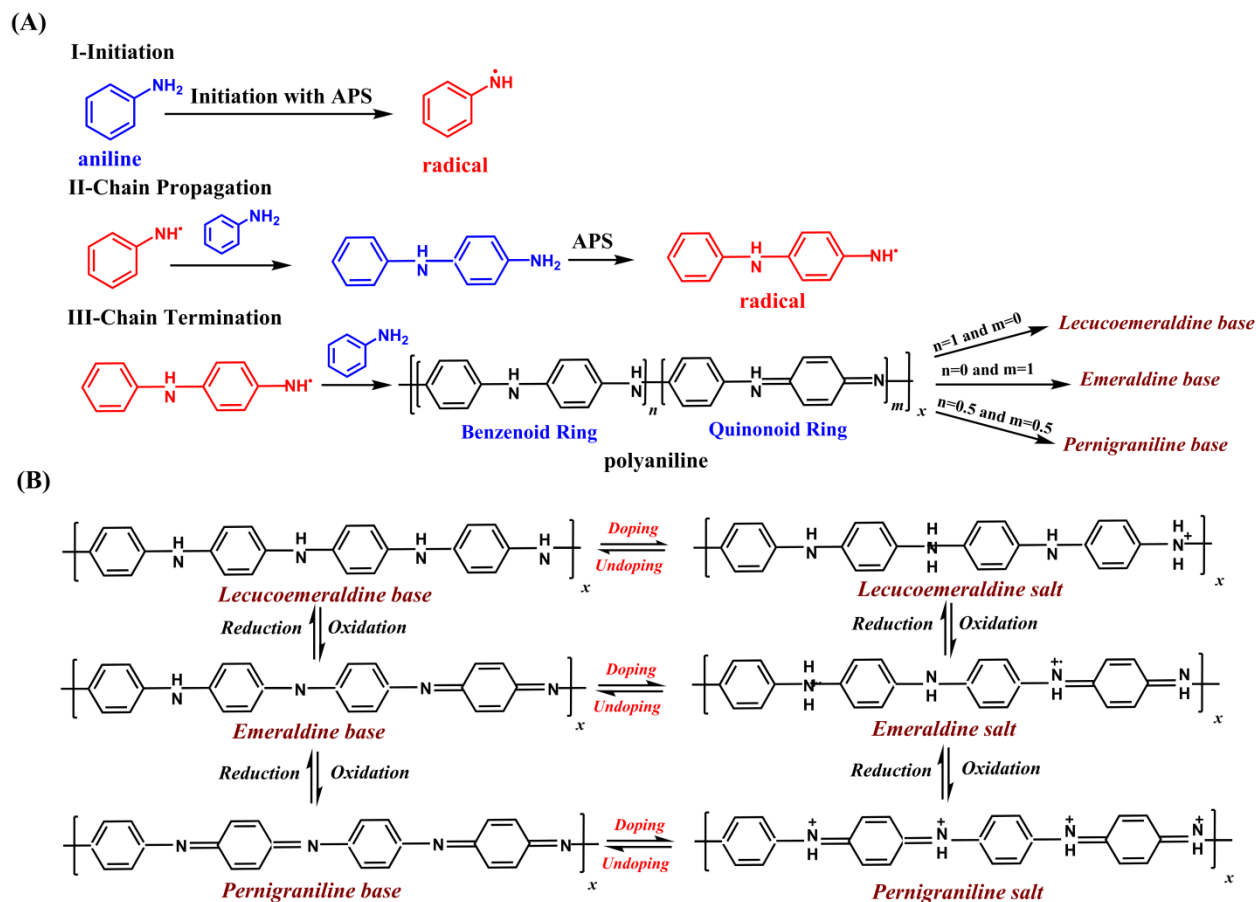


Fig. 3. (A) Reaction mechanism of aniline polymerization through chemical oxidation:  $x = 0, 0.5,$  and  $1$  representing the leucoemeraldine, emeraldine, and pernigraniline forms of PANI, respectively. (B) Different redox forms of PANI with its doped states.

The different tuneable properties of PANI make it a suitable candidate for various applications. Leucoemeraldine is a fully reduced state. Pernigraniline is the fully oxidized state with imine links instead of amine links. Emeraldine base is a neutral form of PANI which can be doped by an acid to produce protonated nitrogen atoms of imine groups. Emeraldine base is regarded as the most useful form of PANI due to its high stability at room temperature and its doped form (emeraldine salt) is electrically conducting. These forms may be inter-converted by chemical and/or electrochemical oxidation or reduction [34]. PANI is a prospective candidate adsorbent for wastewater treatment owing to active amine and imine groups as adsorption sites



interacting with pollutants in aqueous solutions [36]. As shown in Fig. 4, the morphology of PANI is of research interest because of its diverse and tailorable shapes (e.g. nanotube [12], nanofiber [37], nanoflake [38], and nanopowder [39]), pore sizes, pore size distributions, and free volumes [38].

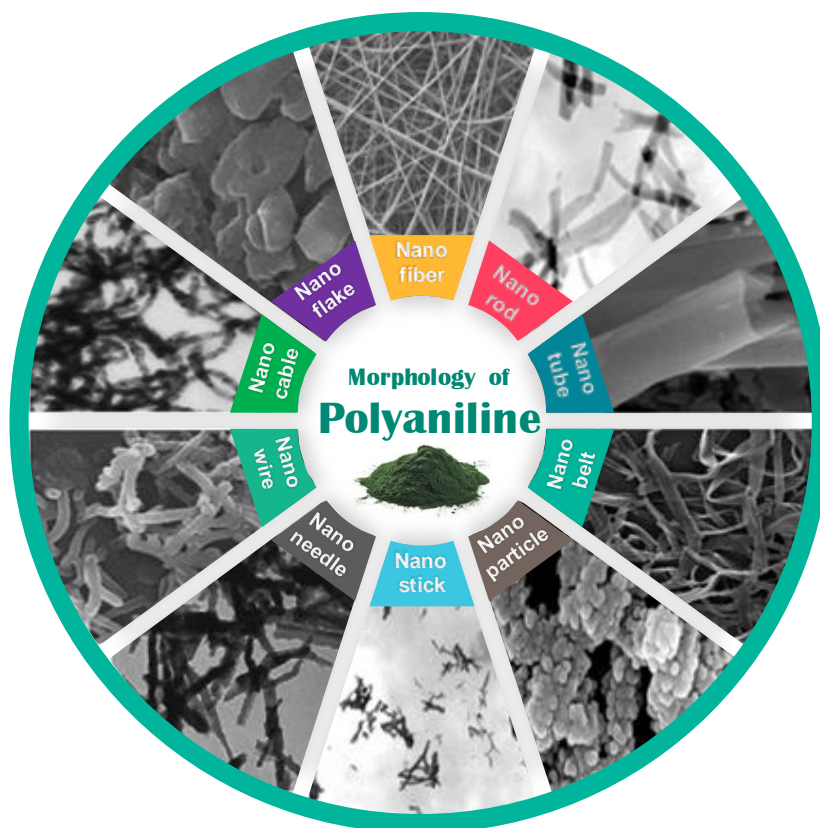
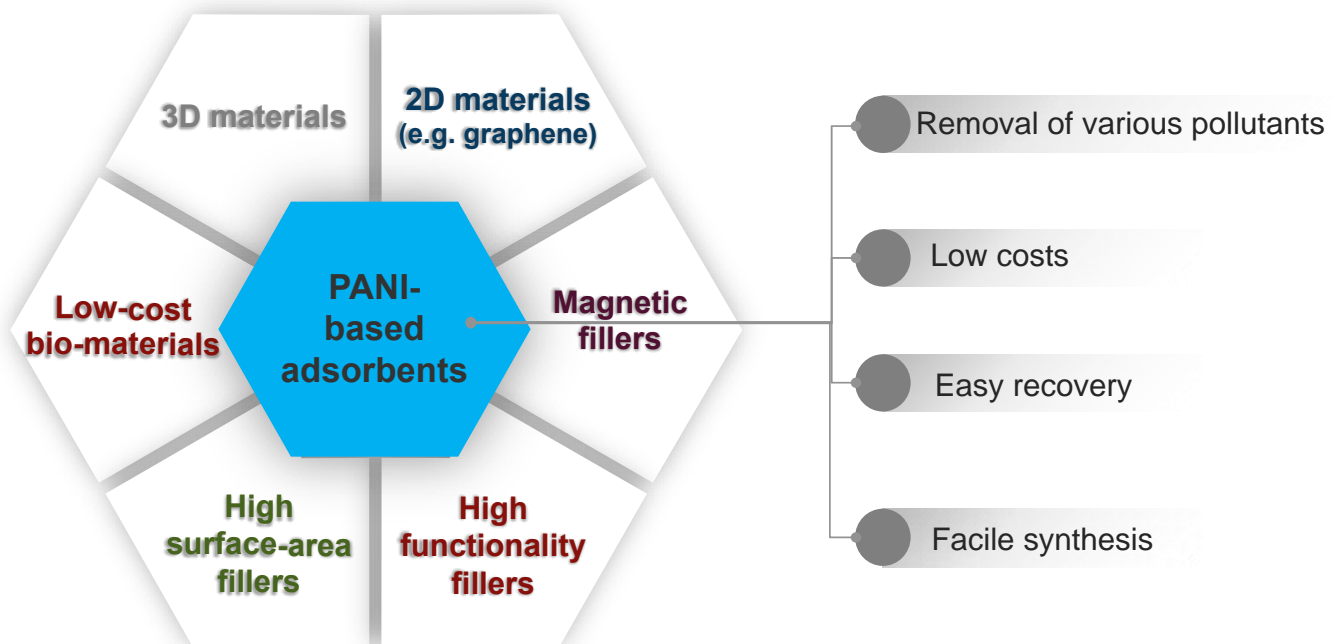


Fig. 4. SEM/TEM images of PANI (a) nanofiber [40], (b) nanotube [41], (c) nanoflake [39], (d) nanoparticle [42], (e) flower-petal [43], (f) nanowire [44], (g) nano-cable [45], (h) nano-needle [46], (i) nano-belt [47], (j) nano-rod [48], (k) nanostick [49].

## 2.2. PANI-based adsorbents

PANI and its derivatives have been widely used in various applications due to their unique characteristics, such as facile synthesis, excellent electrical conductivity, low costs, and good environmental stability [10, 15, 50]. Compared with PANI, its derivatives such as Poly(ortho-phenylenediamine) (PoPDA) have similar chemical structures but different pendant functional groups. PANI and its derivatives have been used as

adsorbents for the removal of various pollutants from drinking water and wastewater due to their large amounts of amine and imine functional groups. PANI derivatives have good solubility in common organic solvents, and they have more functionality but have poor conductivity in comparison with PANI [10]. The forms of pernigraniline and emeraldine exist in either salts or bases. Emeraldine is regarded as the most stable and most useful form of PANI, consisting of equal number of oxidized and reduced units. Imine group of emeraldine base becomes polycationic when doped or protonated with acids. Emeraldine base is widely used as an adsorbent while pernigraniline and its salts can decompose in air because of quinonediimine fragment which is unstable in the presence of a nucleophile, specifically water [15]. To overcome the issues of PANI, enhance its mechanical property/recovery or create synergistic adsorption, PANI has been hybridized with different materials (e.g. clays, carbon nanotubes, and graphene). These PANI-based adsorbents are generally synthesized by in-situ polymerization of aniline. Various PANI-based adsorbents combining different features of the fillers and their key advantages are summarized in Fig. 5.



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Fig. 5. Diverse types of PANI-based adsorbents with different fillers and their key advantages.

### 2.3. Synthesis and characterization of PANI-based adsorbents

PANI can be synthesized by chemical oxidation [51] or electrochemical oxidation [52] polymerization of aniline. PANI is more often synthesized by chemical oxidative polymerization of the aniline via free radical initiators, such as ammonium peroxydisulfate, potassium persulfate, ferric chloride, potassium dichromate, and chloroaurate as oxidants [13, 53-57]. Typical chemical polymerization routes for the synthesis of PANI and PANI-based adsorbents are depicted in Fig. 6. Chemical polymerization of aniline via oxidation includes three main steps: initiation, chain propagation and termination with a radical-dominant mechanism (Fig. 3A) [8, 58]. Electrochemical polymerization can be realized by employing one of the three techniques: (1) applying a constant current (galvanostatic), (2) applying a constant potential (potentiostatic), or (3) applying a potential scanning/cycling to the aqueous solution of aniline [7, 15]. Electrochemical polymerization is typically performed in a strongly acidic aqueous electrolyte by a radical polymerization mechanism, thereby forming anilinium radical cations by aniline oxidation on the electrode [8]. The main approach for the synthesis of PANI-based adsorbents is in-situ polymerization in the presence of organic/inorganic fillers (Fig. 6B), leading to enhanced distribution of the filler and low agglomeration of PANI molecules [55, 59-68].

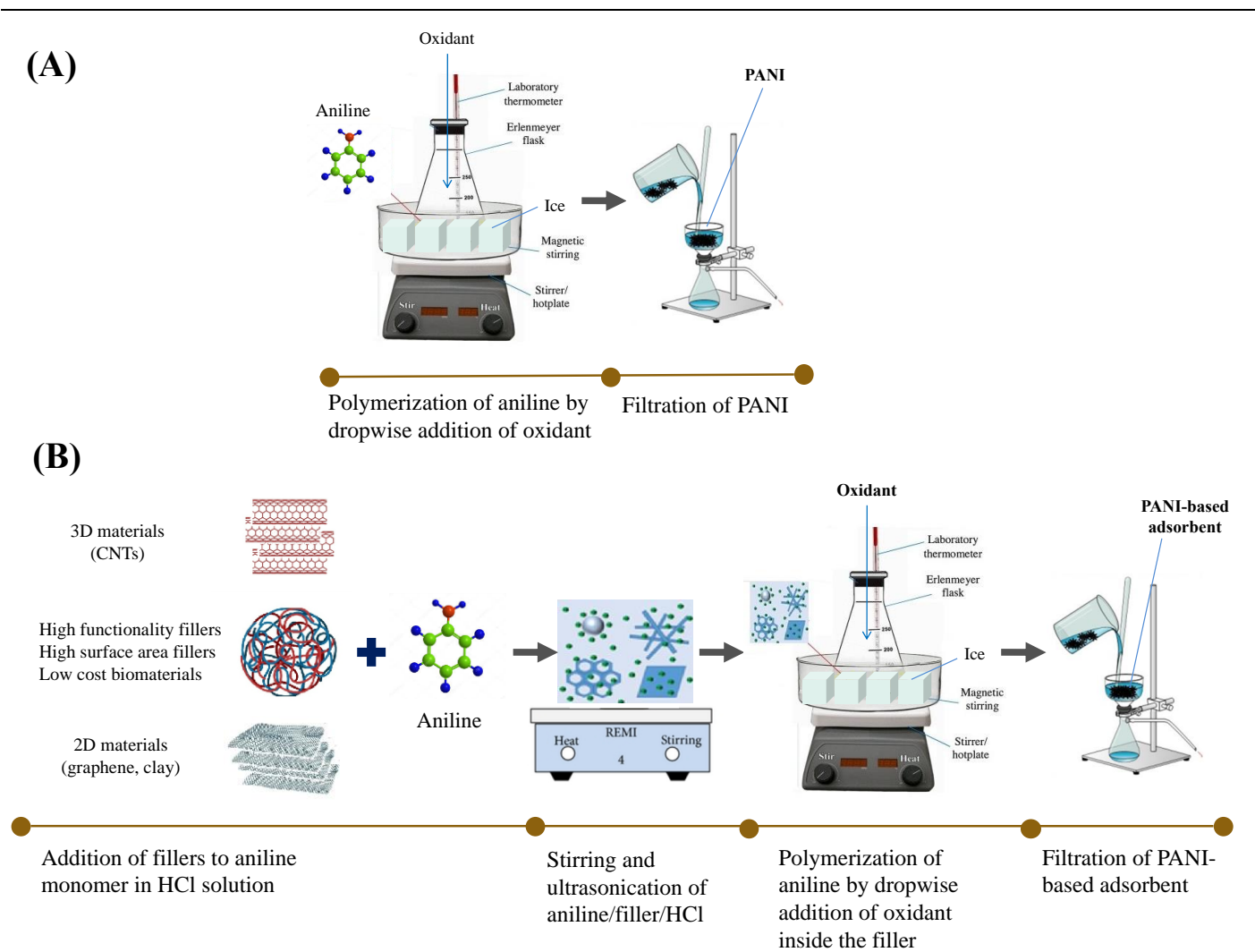


Fig. 6. Preparation of (A) PANI and (B) PANI-based adsorbents by chemical polymerization of aniline.

The morphology of PANI is of great interest because of its diverse and tailorable shapes (e.g. nanotube [12], nanofiber [37], nanoflake [38], and nanopowder [39]), pore sizes, pore size distributions, and free volumes [38]. Compared with a single adsorbent, the PANI composite adsorbent often has enhanced adsorption capability, likely due to the increased surface area and pore volumes of the composite [11]. Samadi et al. synthesized PANI/clay nanocomposites with flower-petal morphology through in-situ polymerization of aniline in the presence of organophilic clay (Cloisite 30B). Study of the chemical structure and morphology of PANI/clay nanopowder by Fourier-transform infrared spectroscopy (FTIR), X-ray powder diffraction (XRD), and scanning electron microscope (SEM) showed exfoliation of the silicate layers throughout the

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PANI matrix [69].

FTIR results provide information about specific functional groups, existence of any impurities or remained solvents. Xu et al determined amounts of the remaining camphor sulfonic acid in the synthesized polyaniline nanotubes by relating the bands at 1037 and 501  $\text{cm}^{-1}$  to the absorption of  $-\text{SO}_3\text{H}$  groups from camphor sulfonic acid [13]. FTIR data before and after adsorption also give evidence on adsorption. The adsorption band at 1240  $\text{cm}^{-1}$  corresponding to the C-F with higher intensity after adsorbing perfluorooctanoate (PFOA) and perfluorooctane sulfonate (PFOS), suggesting their adsorption by the PANI nanotubes [13]. Shifts in the wavenumber of the peak also give some clues on the possible interactions between the adsorbent and the filler. X-ray photoelectron spectroscopy (XPS) spectra, in combination with FTIR, provide useful information on the adsorption of various substances on the surface of the adsorbent. The variation of the detected functional groups and the charge of metal ions play an important role in the adsorption mechanism [70].

Surface areas and pore volumes are important factors for the adsorption capacities of PANI-based adsorbents and they are generally measured by  $\text{N}_2$  adsorption-desorption using Brunauer-Emmett-Teller (BET) analysis. Incorporation of fillers in the PANI matrix or penetration of PANI into 2D and 3D structures of the fillers often leads to increased surface areas of the adsorbents and thus enhanced adsorption capacities [71-75]. For example, Liu et al. prepared PANI/emulsion microsphere composites for adsorption of methyl orange and found that the modification of PANI with mono-dispersed poly(styrene-methyl methacrylate-acrylic acid) microspheres increased the surface area of PANI from 22.8 to 41.7  $\text{m}^2/\text{g}$  and also the adsorption capacity from 140 to 155  $\text{mg}/\text{g}$  [76]. In another work, Dutta et al. prepared nanofibers of PANI hollow microsphere/ $\text{Fe}_3\text{O}_4$  nanocomposite with a surface area of 64  $\text{m}^2/\text{g}$ , which was higher than PANI and conventional PANI/ $\text{Fe}_3\text{O}_4$  composite [74]. Some examples of PANI-based adsorbents with enhanced surface areas are presented in Table 1.

Table 1. BET surface areas and pore volumes of PANI-based adsorbents.

Adsorbents	Surface area (m <sup>2</sup> /g)	Pore volume (cm <sup>3</sup> /g)	Pore diameter (nm)	Pollutants	References
CuFe <sub>2</sub> O <sub>4</sub> /PANI	30.8	0.06	17.8	Hg(II)	[71]
PANI/clay	26.32	0.12	17.55	Acid Green 25	[72]
PANI	18.28	0.07	15.63		
PANI/PSMA	41.7	-	-	methyl orange	[76]
PANI	22.8	-	-		
GO/SiO <sub>2</sub> @PANI	150.36	0.07	9.32	Cu(II), Cr(VI)	[73]
PNHM/Fe <sub>3</sub> O <sub>4</sub>	64	0.16	10.3	As(III)	[74]
PANI/Clay	182.11	1.6	3.8	Cu(II)	[75]
PANI nanofiber	48.83	-	-	methyl orange	[77]
PANI	13.65	-	-		
PANI/Fe <sub>3</sub> O <sub>4</sub>	99.8	0.06	3	Basic Blue 3	[11]
PANI	46.7	0.02	3		
PANI/BiVO <sub>4</sub>	23.53	-	-	methyl orange	[78]
PANI	9.14	-	-		

PSMA: poly(styrene-methyl methacrylate-acrylic acid); PNHM: PANI hollow microsphere.

### 3. Typical aqueous pollutants removed by polyaniline-based adsorbents

PANI-based adsorbents have been applied for the removal of a wide variety of aqueous pollutants. These pollutants can be primarily categorized into: (1) (inorganic) heavy metals and metalloids, (2) (organic) dyes, and (3) emerging pollutants. This section outlines the pollutants and their removal by PANI-based adsorbents.

#### 3.1. Heavy metals and metalloids

Heavy metals (e.g. arsenic, copper, zinc, nickel, cadmium, chromium, mercury, and lead) generated from various industries, such as mining, metal plating, tanneries, batteries, fertilizers, and pesticides, can cause detrimental environmental problems and health issues. Although conventional adsorbents, such as silica, clay minerals, and zeolites have been used for the removal of heavy metals, they face the issues of high costs and/or low efficiency during the the treatment and disposal of the residual metal sludge [79]. Polymer

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nanocomposites are promising adsorbents for the removal of metal ions due to their intrinsic dimensionality, and textural and chemical properties (e.g. surface area, pore-volume, and pore diameter). In particular, PANI-based adsorbents are excellent candidates, as PANI has highly reactive adsorption sites (-NH) with enhanced adsorptive properties. Furthermore, PANI has amphoteric carbonaceous surface groups that can be easily protonated (positively charged) or deprotonated (negatively charged) depending on the solution pH. Chelating groups, such as sulfur, oxygen, and phosphorus can also be added to PANI to chemically interact with charged metal ions [79].

PANI-based adsorbents, including PANI, PANI derivatives, and PANI composites with other materials, have been widely employed for the removal of various heavy metals and metalloids (Table 2), such as Cr(VI) [80], Hg(II) [81], Cu(II) [73, 82], Cd(II) [83], As(III) [74], Pb(II) [84], and Zn(II) [85]. Unique chemical nature, surface area, morphology, and diverse functional groups are the most important properties of these adsorbents which often have excellent adsorption capacities for heavy metals and metalloids. Fig. 7 shows the different morphologies of the synthesized PANI derivatives based on phenylenediamine (i.e., PoPDA, PpPDA: poly(para- phenylenediamine), and PmPDA: poly(meta- phenylenediamine)) [10]. The morphology and chemical structure of the adsorbent significantly affect the adsorption capacity. For instance, the maximum Pb(II) adsorption on the PANI derivatives is in the order of: PpPDA microspheres > PmPDA microparticles > PoPDA hollow microspheres [10].

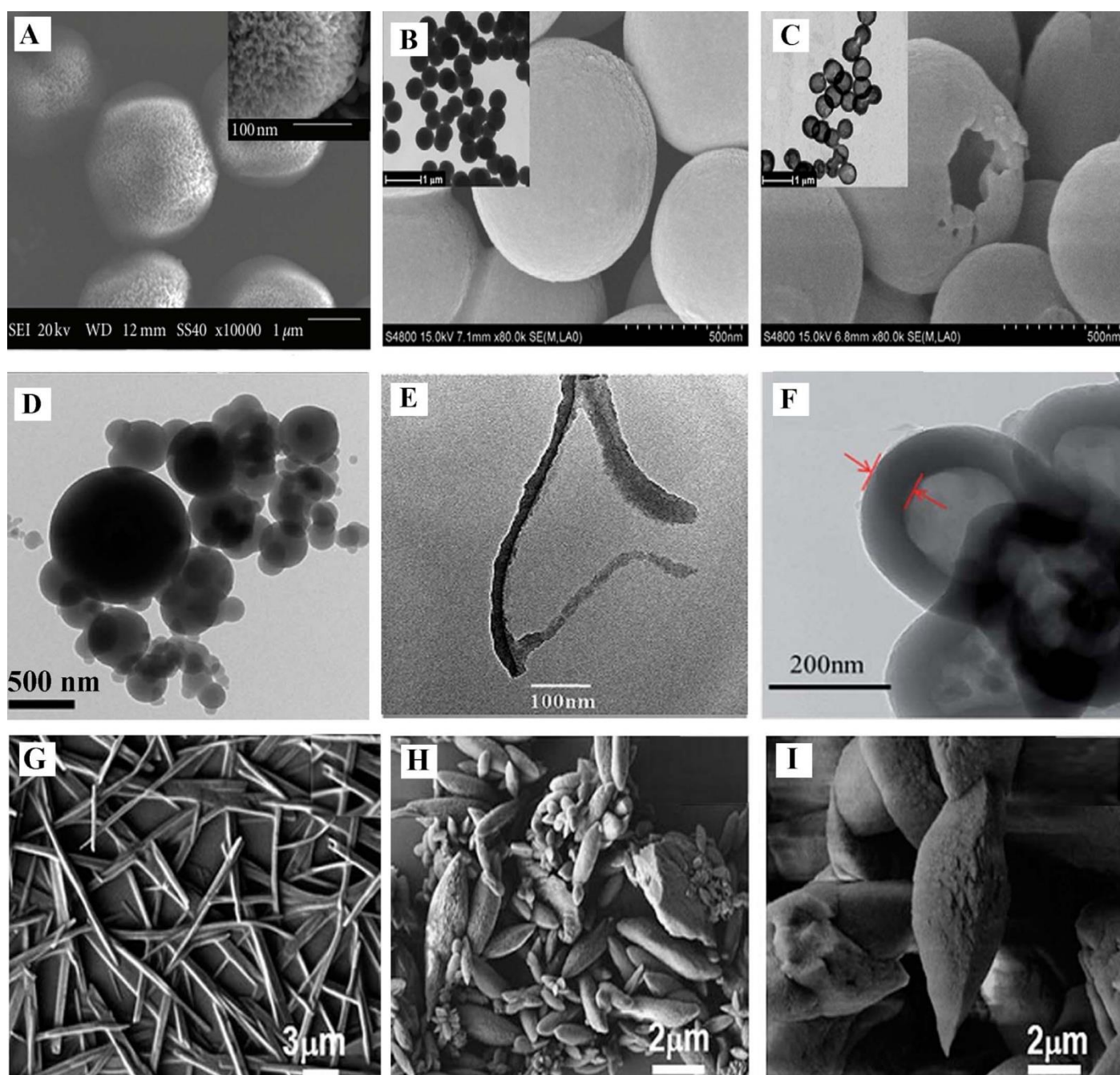


Fig. 7. SEM images of the PoPDA hollow microspheres (A, B and C) and PpPDA microstructures (G, H and I) and TEM images of the PmPDA microstructures (D, E and F) synthesized by different methods [10].

During the adsorption of heavy metal ions by PANI-based adsorbents, metal ions form complexes with the oxygen-, nitrogen-, sulfur-, or phosphorus-containing groups of the incorporated ligands, which act as chelating agents and provide a negative residual charge to the surface of the composites [79]. The chelating mechanism is affected by various parameters, such as pH, ionic strength, number of functional groups, and



the presence of natural organic matters. The competitive adsorption between electrolytes and metal ions can be altered by ionic strength via affecting the electric double layer of hydrated particles, thereby changing the way of metal ions binding with the chelating groups. In addition, low pH is desirable for adsorption of lead, zinc, cadmium, and chromium ions since they would precipitate at high pH values. The removal of metal anions, such as Cr(VI) by PANI-based adsorbents is more efficient at lower pH levels because of the stronger electrostatic attraction between aqueous Cr(VI) oxyanions and protonated imine groups [16]. A schematic representation of the adsorption of metal ions by PANI-based adsorbents and the possible adsorption mechanisms are illustrated in Fig. 8.

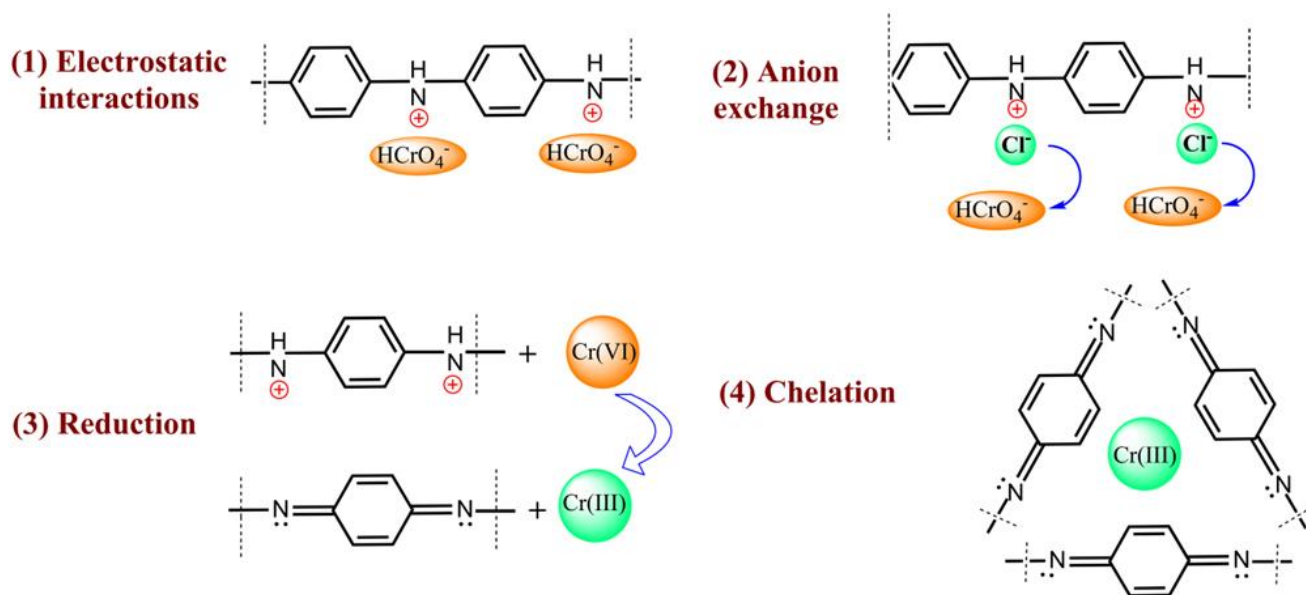


Fig. 8. Proposed mechanisms of metal ions removal by PANI-based adsorbents using Cr(VI) as an example, including several steps: (1) electrostatic interactions between the negatively charged aqueous Cr(VI) oxyanions and protonated imine groups, or (2) ion exchange between doped aqueous Cr(VI) oxyanions and anion (e.g. Cl<sup>-</sup>) doped in PANI. Step (1) or (2) leads to Cr(VI) adsorption onto the PANI surface. (3) Some Cr(VI) oxyanions are reduced into Cr(III) cations while amines are oxidized into quinoid amine. (4) The oxidized quinoid amines of PANI are bound with partial Cr(III) cations.

The variation of the functional groups of the adsorbents and the charge of metal ions play an important role in

the adsorption mechanism [70]. Generally, electrostatic interaction, participation, reduction, complexation, and ion-exchange are the main mechanisms of metal ion adsorption by PANI-based adsorbents. Kumar et al. investigated the binding between Cu(II) and Cr(VI) ions with graphene oxide (GO)/SiO<sub>2</sub>@PANI composite using FTIR and XPS analysis [73]. By analyzing FTIR peaks of SiO<sub>2</sub> functional groups, they found a compressed SiO<sub>2</sub> network in GO/SiO<sub>2</sub>@PANI. All the observed bands of GO/SiO<sub>2</sub>@PANI after Cu(II) and Cr(VI) binding slightly shifted due to the interaction with metal ions. The peaks for -OH, -NH, -NH<sub>2</sub>, -COOH groups moved to low intensity, revealing the role of these groups in the adsorption of Cu(II) as well as Cr(VI) onto GO/SiO<sub>2</sub>@PANI. The slight change in the binding energies and the occurrence of new peaks from XPS data confirmed the interaction between the metal ions and GO/SiO<sub>2</sub>@PANI composite. The highest sequestering of Cu(II) was observed at pH 5.3 (i.e. >pH<sub>zpc</sub>) due to electrostatic interaction between the deprotonated hydroxyl, carboxylic, and amine groups of the GO/SiO<sub>2</sub>@PANI. Apart from the electrostatic interaction and complexation, adsorption of Cu(II) may also be caused by the cation- $\pi$  interaction. The GO/SiO<sub>2</sub>@PANI contains the electron-rich domains on the aromatic rings, and Cu(II) can bind with GO/SiO<sub>2</sub>@PANI *via* cation- $\pi$  interaction. The adsorption reduction behavior of Cr(VI) to Cr(III) can be explained via a multiple stage mechanism: (i) at the optimum pH (pH 3) for the removal of Cr(IV), Cr(VI) adsorbs electrostatically on the active sites (e.g. -N<sup>+</sup> and -OH<sub>2</sub><sup>+</sup>) of GO/SiO<sub>2</sub>@PANI; (ii) electron rich N containing functional groups reduce Cr(VI) to Cr (III); and (iii) deprotonated -N- groups of PANI, carboxylic and hydroxyl groups of the GO and SiO<sub>2</sub> adsorb Cr (III) [73].

Table 2. Adsorption of inorganic heavy metals and metalloids by PANI-based adsorbents.

PANI-based adsorbents	T (°C)	pH	Pollutants	Q (mg/g)	References
<b>PANI polymer</b>					
PANI particles	25	7	Cr(VI)	-	[86]
PANI nanosheets	25	2	Cr(VI)	263.2	[54]
<b>PANI derivatives</b>					

<b>PANI-based adsorbents</b>	<b>T (°C)</b>	<b>pH</b>	<b>Pollutants</b>	<b>Q (mg/g)</b>	<b>References</b>
PpPDA microspheres	25	-	Pb(II)	> 1800	[92]
MnO <sub>2</sub> @PmPDA	25	5.4	Pb(II)	367	[91]
PoPDA hollow microspheres	30	6	Pb(II)	103.2	[87]
PmPDA/Fe <sub>3</sub> O <sub>4</sub> composite	25	1-13	Cr(VI)	2750	[93]
PmPDA-polypyrrole	25	2	Cr(VI)	526	[90]
PmPDA-Fe <sub>3</sub> O <sub>4</sub>	15-45	2-9	Cr(VI)	246.1	[89]
Hollow Cu-PmPDA particles	35	5.2	As(V)	27.4	[88]
<b>PANI copolymers</b>					
pyrrole	25	2	Cr(VI)	227	[95]
1,8-diaminonaphthalene	25	4	Cr(VI)	150	[94]
PANI grafted chitosan	25	6	Pb(II)	3.2	[96]
m-phenylenediamine	25	6	Cd(II)	-	[97]
<b>PANI-modified mats and membranes</b>					
Polyacrylonitrile mat	25	2	Cr(VI)	52	[98]
Cellulose acetate membrane	30	5	Hg(II)	280	[99]
Polystyrene mat	25	4	Pb(II)	312	[100]
Poly(ethylene-co-vinyl alcohol) mat	20	2	Cr(VI)	93.1	[101]
Polyimide membrane	25	1	Cr(VI)	48.9	[102]
<b>Synthetic polymers</b>					
Polystyrene	30	4.1	Hg(II)	-	[66]
Polystyrene	30	4	Cr(VI)	19	[103]
PEG	25	5	Cr(VI)	109.9	[104]
PPy	25	2	Cr(VI)	227	[68]
Polyvinyl alcohol	50	4	Cr(VI)	111.2	[105]
Polyethylene terephthalate	25	2	Cr(VI)	3.0	[106]
<b>Polysaccharide biopolymers</b>					
Alkyl-substituted PANI/ chitosan	25	4.2	Cr(VI)	229.8	[61]
Chitosan grafted PANI	30	4.2	Cr(VI)	165.6	
PANI/CA	25	3-7	Cu(II)	68.0	[107]
	25	3-7	Pb(II)	251.3	
Chitosan grafted PANI	30	4.2	Cr(VI)	179.2	[108]
PANI/dextrin	25	3	Cu(II)	-	[60]
PANI coated ethyl cellulose	-	1	Cr(VI)	38.8	[109]
PpPDA/chitosan	30	6	Cu(II)	650	[110]
PANI coated chitin	30	2	Cr(VI)	23	[65]
Sodium alginate/PANI	30	2	Cr(VI)	78.63	[80]
<b>Agriculture wastes</b>					
PANI/SD	25	6	Pb(II)	-	[111]
PANI/jute fiber	20	3	Cr(VI)	62.9	[112]
PANI/SD	20	5	Cu(II)	208.8	[113]

<b>PANI-based adsorbents</b>	<b>T (°C)</b>	<b>pH</b>	<b>Pollutants</b>	<b>Q (mg/g)</b>	<b>References</b>
PANI/SD	25	5	Cd(II)	430	[114]
PANI/RHA	25	9	Hg(II)	-	[67]
PANI/RHA	25	3	Zn(II)	24.3	[115]
PANI-kapok fiber	30	4-5	Cr(VI)	66.2	[59]
PANI/SD/PEG	25	<2	Cr(VI)	3.2	[116]
<b>Carbon-based materials</b>					
Multiwalled CNT	25	4.5	Cr(VI)	31.8	[62]
GO	25	5.8	Cu(II)	38	[117]
GO	30	6.5	Cr(VI)	192	[118]
PANI/GO-CNT	25	2	Cr(VI)	142.9	[119]
PANI/GO	25	7	Zn (II)	210	[85]
<b>Metal compounds</b>					
Fe <sub>3</sub> O <sub>4</sub>	25	3	Cr(VI)	6	[120]
Magnetic mesoporous silica	25	8	Cr(VI)	193.9	[64]
Magnetic mesoporous carbon	20	2	Cr(VI)	150	[121]
<b>Clay minerals</b>					
PANI/sepiolite	25	2	Cr(VI)	206.6	[122]
PANI/palygorskite	15	5.8	Cr(VI)	11.5	[63]
PANI/clay	25	5	Pb(II)	7.4	[84]
PANI/Halloysite	25	5.1	Cr(VI)	69.3	[123]
PANI/starch/montmorillonite	25	2	Cr(VI)	208.3	[124]

ES: emeraldine salt; PoPDA: Poly(ortho- phenylenediamine); PmPDA: Poly(meta- phenylenediamine); PpPDA: Poly(para- phenylenediamine); PEG: polyethylene glycol; CA: cellulose acetate; SD: sawdust; RHA: rice husk ash; CNT: carbon nanotube; GO: graphene oxide.

### 3.2. Organic dyes

Wastewater containing dyes mainly come from the textile industry, and annually ~280,000 tons of dyes mixed with saline water are discharged into the environment, posing detrimental impacts on public health and ecological systems [125-127]. The dye is generally described as a colored substance having two key components: chromophores, contributed to the color, and auxochromes, attached to the chromophore and having aromatic molecular structures. Typically, dyes can be classified into cationic (all basic dyes), anionic (direct, acid, and reactive dyes), and non-ionic (disperse dyes). Many efforts have been made to remove dyes

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from water and wastewater as they can cause mutagenesis, chromosomal fractures, carcinogenesis, and respiratory toxicity [10, 15, 128]. As summarized in Table 3, different PANI-based adsorbents have been applied for the removal of a diversity of dyes, such as reactive dyes [129], acid dyes [130], methylene blue [131], methyl orange [77], Orange 5 [132], direct dyes [133], Congo Red [119], and Rhodamine B [134].

Adsorption of dyes by PANI-based adsorbents is influenced by many factors. Functional groups of the adsorbent and adsorbate, and the possible reactions between them, are the key factors determining the effectiveness of the adsorption [10]. Dyes can be cationic, anionic, or non-ionic upon dissolution in aqueous media [10]. Therefore, the reversely charged adsorbents can have electrostatic interactions and chemisorptions with dyes. The charge properties of PANI-based adsorbents can be tailored by altering the surface charge of PANI during doping/dedoping, changing the solution pH, and incorporating fillers with charges opposite to those of the target dyes. Patra et al. investigated the removal of anionic dyes by potash alum doped PANI and reported that the main mechanisms for the dye removal by PANI are the interactions of the positive fragments of the PANI backbone with the negatively charged parts of the dye molecule, and the basic sites of PANI with the positively charged dye [135]. Khadir et al. reported similar electrostatic interactions between the negative  $-\text{SO}_3^-$  group of the anionic dye and the positive groups of a sisal fibers/PPy/PANI bio-adsorbent in the acidic medium as shown in Fig. 9 [129].

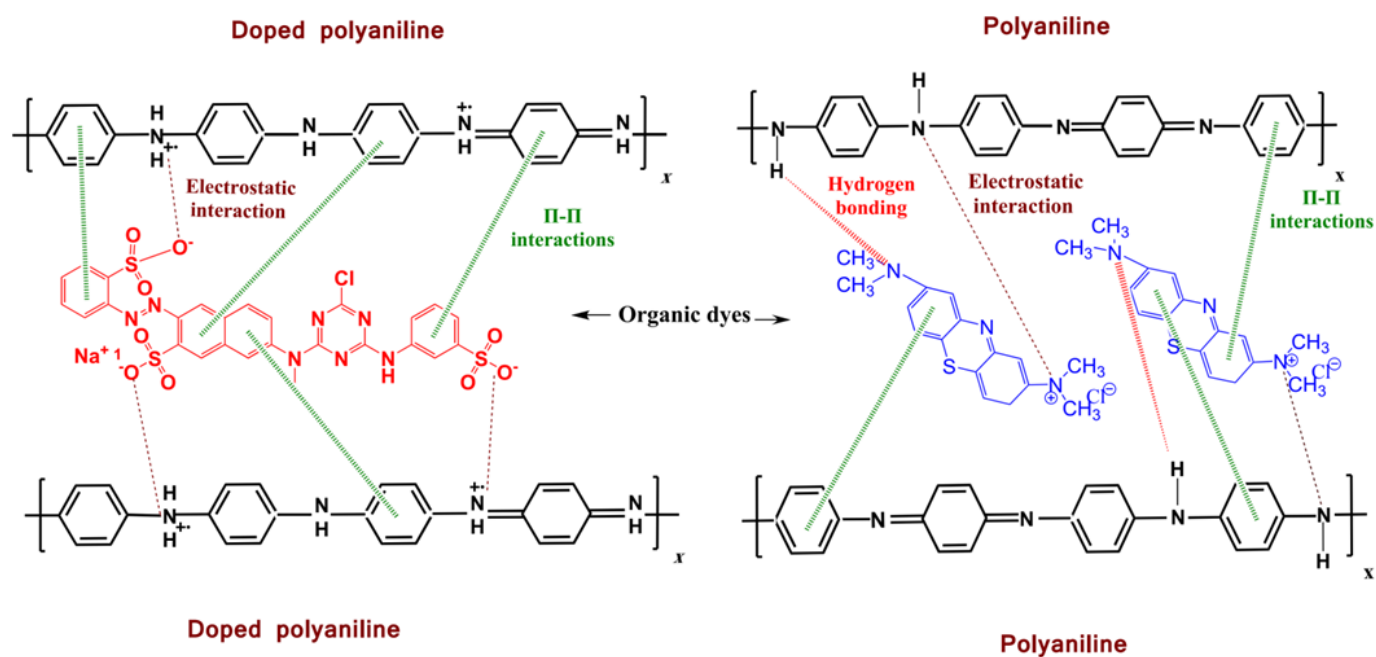


Fig. 9. Schematic illustration of the possible mechanisms (including physical and chemical interactions: electrostatic interaction,  $\pi$ - $\pi$  interaction and hydrogen bonding) in the adsorption of anionic (left) and cationic (right) organic dyes by PANI-based adsorbents.

Moreover, hydrophilic/hydrophobic properties, hydrogen bonding, and dipole-dipole interaction also play important roles in the adsorption process [15]. Compared with a single adsorbent, the PANI composite adsorbent often has enhanced adsorption capability, due to the increased surface areas and pore volumes of the composite [11]. Removal of different dyes by numerous PANI-based nanocomposites, such as PANI/agriculture waste, PANI/biopolymer, PANI/polypyrrole, PANI/polyvinyl alcohol, PANI/polyacid, PANI/silica, PANI/zirconium, PANI/silver, PANI/iron oxide have been examined recently, and the possible adsorption mechanisms (taking the cationic methylene blue dye as an example), including electrostatic attraction,  $\pi$ - $\pi$  interaction, and hydrogen bonding are schematically illustrated in Fig. 9 [15].

Magnetic nanomaterials have attracted growing research interest for dye removal from wastewater. In particular, magnetic mesoporous nanocomposites are composed of a magnetic core with strong magnetic interactions and a polymeric shell with functional groups that maintain adsorbent pore size distribution and

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prevent agglomeration of magnetic metal compounds [16]. A schematic representation of the synthesis and functionalization of the  $\text{Fe}_3\text{O}_4@\text{PANI}$  composite via in-situ polymerization, and adsorption/desorption mechanism of malachite green dye is represented in Fig. 10 [136]. To achieve high adsorption capacity by increasing the surface area, kapok fiber-oriented PANI was prepared by the chemical oxidation method for the removal of sulfonated dyes (Fig. 11), and the monolayer adsorption capacities based on the Langmuir equation were 40.8, 188.7, and 192.3 mg/g for Congo Red, Orange II, and Orange G, respectively [137].

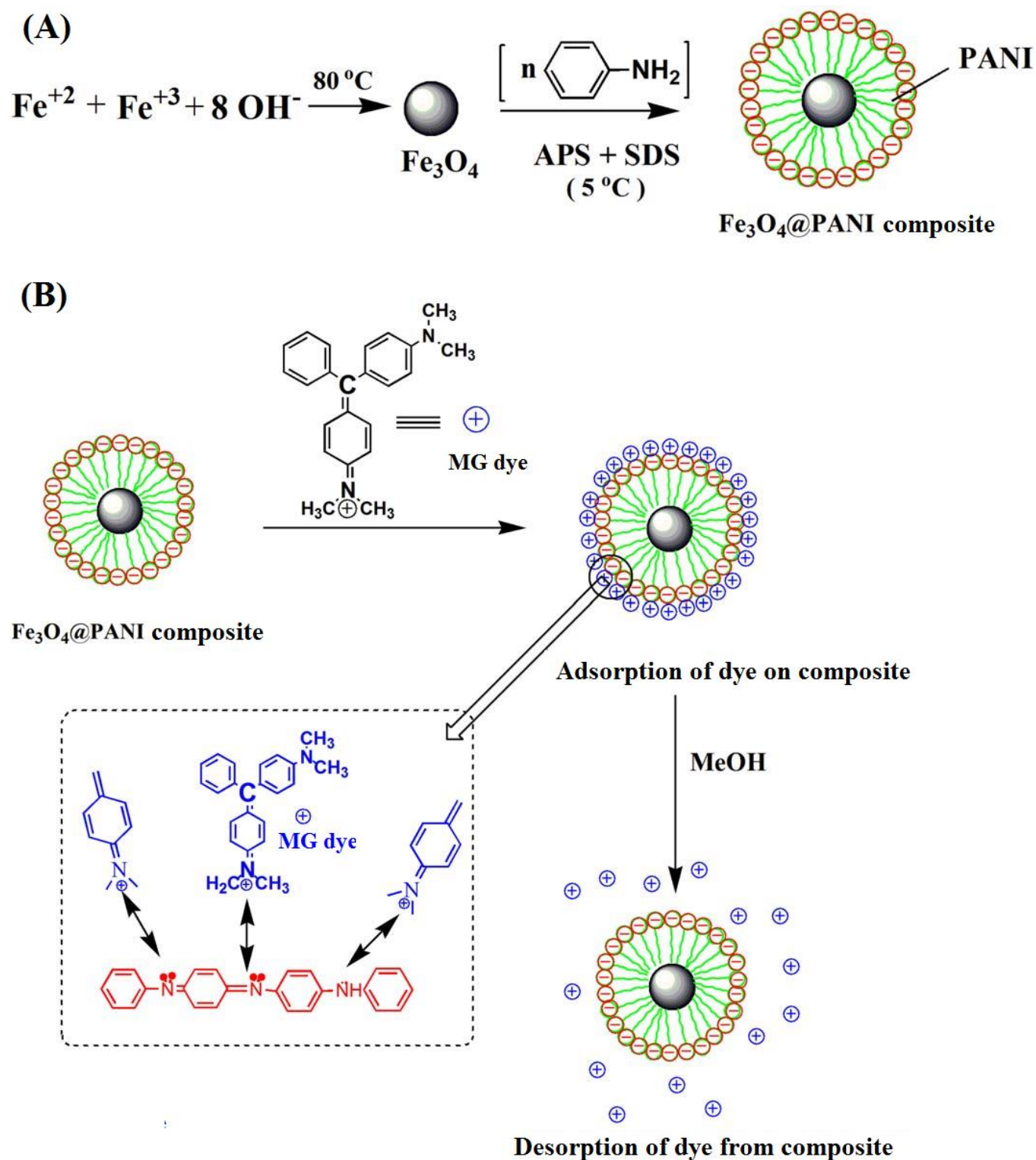


Fig. 10. Schematic representation of (A) the synthesis and functionalization of the  $\text{Fe}_3\text{O}_4$  nanoparticles via in-situ polymerization and (B) adsorption/desorption mechanism of Malachite green dye [136].



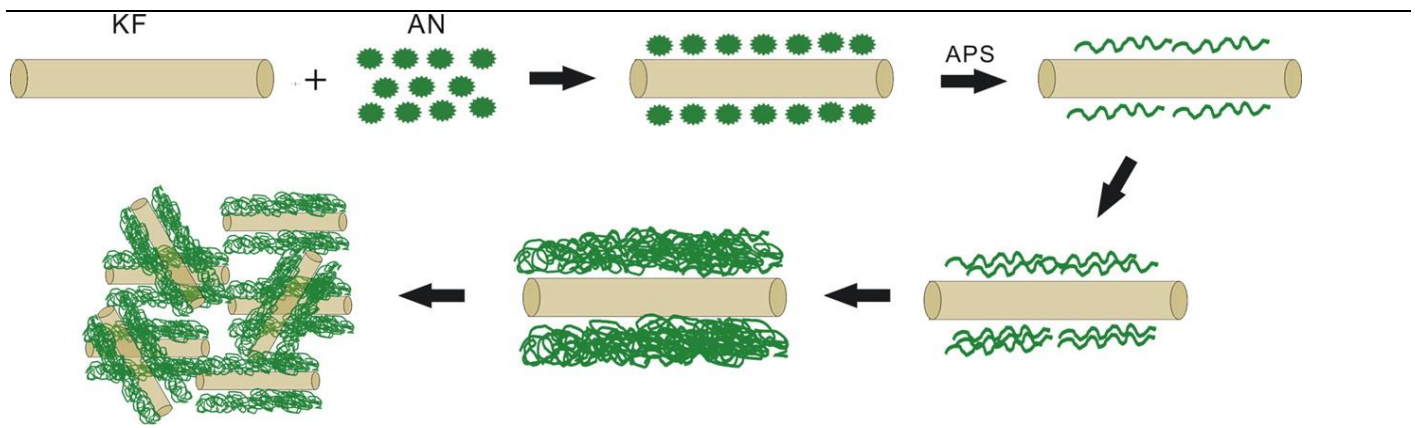


Fig. 11. Schematic illustration for preparing kapok fiber (KF) oriented PANI [137].

Table 3. Adsorption of typical dyes by PANI-based adsorbents.

PANI-based adsorbents	T (°C)	pH	Pollutants	Q (mg/g)	References
<b>PANI polymer</b>					
PANI-ES	25	5	Orange G	-	[56]
PANI-ES	25	3.4	Orange G	-	[138]
PANI nanotubes	25	10.0	Methylene Blue	4.8	[139]
Cross-linked PANI	25	6.5	Methylene Blue		[57]
PANI nanofibers	25	6.0	Reactive black 5	51.02	[140]
<b>PANI derivatives</b>					
Cu-PmPDA microparticles	25	Acidic	Methylene Blue	96.1	[141]
PmPDA nanobelts	30	-	Orange G	460	[142]
<b>Synthetic polymers</b>					
Polyacid	25	7	Methyl Orange	466.5	[143]
Polyacid	25	7	Rhodamine B	440	
Polyamide	40	Basic	Methyl Orange	81.9	[144]
PANI/chitosan	25	3	Congo Red	322.54	[145]
PANI/Starch	25	3	Reactive Black 5	0.81	[146]
PANI/EPS	25	3	Remazol	-	[147]
Starch-MMT/PANI	-	2	Reactive Blue 194	91.74	[148]
Chitosan-PANI/ZnO	-	3.1	Reactive Orange16	476.2	[149]
Chitosan-grafted-PANI	25	2	Acid Red 4	92	[150]
Chitosan/PANI hybrid	25	4	Acid Green 25	240.4	[151]
<b>Agriculture wastes</b>					
PANI-kapok fiber	30	3	Congo Red	40.8	[137]
PANI/HCl-MRH	-	3	Acid Red 18	100	[152]
PANI-lignocellulose	25	4.3	Congo Red	1672.5	[153]
kapok paper/zinc oxide/PANI	-		Methyl orange	-	[154]
PANI-kapok fiber	25	4.3	Methyl orange	75.76	[82]
<b>Metal compounds</b>					
Nickel ferrite nanoparticle	25	-	Basic Blue 41	0.50	[155]
Fe <sub>3</sub> O <sub>4</sub>	30	7	Malachite Green	4.82	[156]

PANI-based adsorbents	T (°C)	pH	Pollutants	Q (mg/g)	References
NiFe <sub>2</sub> O <sub>4</sub>	30	3.0-4.4	Alizarin Red S	186	[157]
Zinc ferrite	25	2	Rhodamine B	1000	[134]
<b>Clay minerals</b>					
PANI/Beidellite	25	3	Acid Yellow 194	123	[130]
PANI/Clinoptilolite	30	2	Acid Violet 90	72.46	[158]
PANI/montmorillonite	20-50	2-6	Acid Green 25	49.3-49.8	[72]

ES: emeraldine salt; PmPDA: poly(meta- phenylenediamine); EPS: extracellular polymeric substance; MMT: montmorillonite; MRH: modified rice husk.

### 3.3. Emerging pollutants

Emerging pollutants (EPs), are manufactured or man-made chemicals or materials that can probably alter the metabolism of living ecosystems due to their toxicity and persistence [159, 160]. The main EPs include pharmaceuticals, personal care products (PCPs), stimulants, industrial additives, and agrochemicals (e.g. pesticides and herbicides) as summarized in Fig. 12 [161]. EPs widely exist in wastewater, surface water, groundwater, and drinking water, and pose great risks to human health and natural ecosystems, such as hormonal interference in aquatic animals, genotoxicity, endocrine disruption, and immune toxicity [24].

For metal ions, electrostatic interaction is the main driving force for adsorption; while for EPs, as most of them are organic compounds with complex molecular structures, multiple interactions may exist between the adsorbents and the pollutants. In particular, many EPs, such as pharmaceuticals have aromatic and aliphatic structures, which means that  $\pi$ - $\pi$  interactions and hydrophobic interactions will also play important roles in their adsorption by PANI-based adsorbents. In terms of electrostatic interaction similar to the case of metal ions, positively charged adsorbates are more likely to be removed by negatively charged adsorbents and vice versa. Moreover, PANI is a conducting polymer and its electric charge can be easily changed by doping/dedoping or changing the solution pH [162]. The zeta potential analysis is used to evaluate the charge stability of the adsorbent/water system. Negative CuFe<sub>2</sub>O<sub>4</sub>/PANI composite has net charges at pH > 6.0 and

positive charges at  $\text{pH} < 6.0$ . At low pH values (e.g.,  $\text{pH} < 5.0$  for PANI-HCl), nitrogen atoms of imine groups were bound by protons, causing the PANI surfaces carrying positive charges [71]. This would lead to effective removal of negative pollutants, such as PFASs [13, 163].

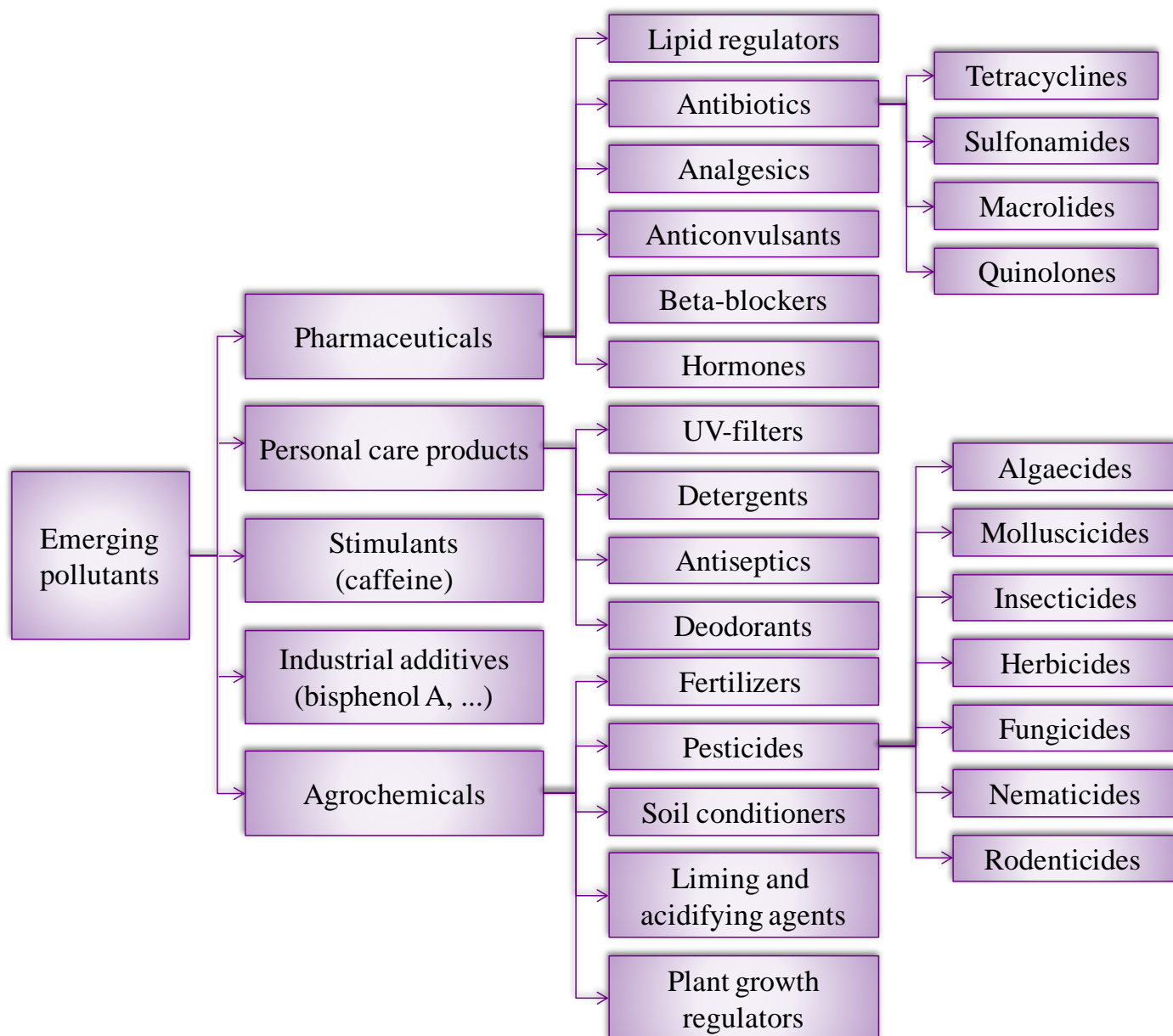


Fig. 12. Main categories and examples of emerging contaminants, modified from [161].

Structural similarity (molecular weight or size) between EPs and dye molecules makes them suitable for sorptive removal by PANI-based adsorbents [5]. Recently, several studies have been reported on the removal of EPs, such as drugs [164], insecticides [165], bisphenol A (BPA) [166], personal care products [167], PFASs

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[168], PCBs [169], endocrine disrupting compounds (EDCs) [170] by PANI-based adsorbents. Xu et al. synthesized PANI nanotubes for the removal of perflourooctane sulfonate (PFOS) and perfluorooctanoate (PFOA) from aqueous solutions [13]. The adsorption isotherms of PFOS and PFOA on the PANI nanotubes fitted well to the Langmuir model, suggesting the monolayer adsorption behavior. The adsorption capacities were 1651 and 1100 mg/g for PFOS and PFOA, respectively, after 48h reaching equilibrium adsorption. They also reported that electrostatic attraction played an important role in the adsorption and other forces such as hydrophobic interaction might be also involved.

Zhou et al. synthesized PANI coated  $\text{Fe}_3\text{O}_4$  ( $\text{Fe}_3\text{O}_4/\text{PANI}$ ) for the removal of BPA, a-naphthol, and b-naphthol and reported adsorption capacity of 23.09 mg/g for BPA at 298 K [171]. In another study, Laabd et al. employed an efficient recyclable PANI/PPy copolymer for the removal of personal care products hemimellitic, trimellitic, and pyromellitic acids from aqueous solution [167]. They found that spontaneous and endothermic adsorption was well described by the pseudo-second-order kinetic model and the Langmuir isotherm model with the maximum adsorbed quantities of 79.94, 93.55, and 111.29 mg/g for hemimellitic, trimellitic, and pyromellitic acids, respectively. They suggested that physisorption of carboxylic groups of the pollutants on the hydrogen atoms in amine groups was the main adsorption mechanism. The main adsorption mechanism between organic contaminants and the PANI-based adsorbent was repellent or attractive electrostatic interactions depending on the charge densities of the adsorbate and the adsorbent which were influenced by the solution pH [172]. Thus, similar to the adsorption of organic dyes and heavy metals, solution pH strongly affects the adsorption process and must be adjusted to provide an appropriate surface charge for the adsorption. Hlekelele et al. synthesized PANI/PPy and zero-valent iron-based materials for the removal of BPA from aqueous solution and found that surface-bound hydroxyl radicals were responsible for the degradation of BPA [172]. They investigated the effect of pH and reported that at high pH values, the surface of the adsorbent

accumulates a negative charge which is repellent to the negatively charged anions that BPA exists as at high pH values. Therefore, electrostatic interactions were part of the adsorption mechanism. It was also found that PPy/PANI was a relatively hydrophobic material at pH 6.2, and BPA was also hydrophobic, meaning that the hydrophobic non-electrostatic interactions were also responsible for the adsorption of BPA onto PPy/PANI. The data of recently published studies on the adsorption of emerging pollutants by PANI-based adsorbents are given in Table 4.

Table 4. Removal of emerging pollutants by PANI-based adsorbents.

PANI-based adsorbents	T (°C)	pH	Pollutants	Q (mg/g)	References
PANI base nanotubes	25	2	Perfluorooctane sulfonate	1651	[13]
	25	2	Perfluorooctanoate	1100	
Fe <sub>3</sub> O <sub>4</sub> / PANI core-shell	25	7	Bisphenol A	23.1	[171]
	25	7	$\alpha$ -naphthol	28.7	
	25	7	$\beta$ -naphthol	9.1	
PANI/magnetic GO	25	6	Ciprofloxacin	89.68	[173]
	35	6	Ciprofloxacin	94.43	
	45	6	Ciprofloxacin	97.78	
PANI derived porous carbon	25	7	Triclosan	1318	[174]
	25	7	Oxybenzone	906	
	25	7	p-chloro-m-xyleneol	840	
PANI/PPy copolymer	20	6	Hemimellitic acid	79.94	[167]
	20	6	Trimellitic acid	93.55	
	20	6	Pyromellitic acid	111.29	
PANI/PPy-Fe <sup>0</sup>	25	< 6	Bisphenol A	90	[172]
PANI-derived carbon	25	7	Atrazine herbicides	943	[175]
	25	7	Diuron herbicides	884	
PANI/PPy	25	6.2	Metronidazole	65	[176]
PANI/peanut husk	35	3	Imidacloprid insecticide	7	[165]

#### 4. Adsorption kinetics and desorption

The study of the adsorption kinetics via diffusion models and reaction models is of great significance to determine the adsorption performance and gain insights into the adsorption mechanisms. The diffusion models propose three steps of film diffusion, intraparticle diffusion, and adsorption-desorption on the active sites

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[177]. Conversely, reaction models, derived from chemical reaction kinetics without considering the diffusion steps are widely used for the removal of aqueous pollutants by PANI based adsorbents [75, 157]. The pseudo-first-order (PFO) model (Eq. 1), also called the Lagergren kinetic model, is the earliest equation for evaluating adsorption mechanisms and potential rate-controlling steps [178].

$$\ln(q_e - q_t) = \ln q_e - Kt \quad (1)$$

where  $K$  is the rate constant of PFO adsorption ( $\text{min}^{-1}$ );  $q_e$  and  $q_t$  are the adsorption capacity at equilibrium and at time  $t$ , respectively ( $\text{mg}\cdot\text{g}^{-1}$ ). However, the pseudo-second-order (PSO) model (Eq. 2) has been more widely used for adsorption:

$$\frac{t}{q_t} = \frac{1}{K_2 q_e^2} + \frac{t}{q_e} \quad (2)$$

where  $K_2$  ( $\text{g}\cdot\text{mg}^{-1}\cdot\text{min}^{-1}$ ) is the rate constant of PSO adsorption.

The main difference of these most frequently used models is that the PFO model does not represent the effects of the adsorbent concentration or the available active sites on the adsorption process. Most adsorption studies have preferred the PSO model more than the PFO model as the former has more accurate predictions under the equilibrium state and complexity of the adsorbent surface as well as the good fitting to linear regression [179].

Determination of the rate-controlling step of the adsorption is very important in exploring the adsorption mechanism and designing an adsorption process [180]. Weber and Morris introduced the intraparticle diffusion model [181]:

$$q_t = K_{id} t^{0.5} + C \quad (3)$$

where  $K_{id}$  is the rate constant and  $C$  is a constant. The slope of the plot of  $q_t$  vs.  $t^{0.5}$  gives the  $K_{id}$  value. In some

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cases, multi-linearity may be observed, suggesting more than one rate controlling steps. The term C is expected to be directly proportional to the boundary layer thickness [167]. A sharp slope may be seen at the first time interval due to the external diffusion [182, 183]. The intraparticle diffusion model has been widely used for adsorption kinetics of PANI-based adsorbents for aqueous pollutants [184-186]. However, for an accurate application of this equation, the adsorption data should be carefully divided into 2-3 time intervals and then each step fits individually [157, 183]. When the PANI-based adsorbents are highly heterogeneous, the Elovich model can be used to determine the chemisorption by [187]:

$$q_t = \frac{1}{\beta} \ln(\alpha\beta) + \frac{1}{\beta} \ln(t) \quad (4)$$

where  $\alpha$  is the initial adsorption rate constant ( $\text{g}\cdot\text{mg}^{-1}\text{min}^{-1}$ ) and  $\beta$  is the desorption rate constant ( $\text{g}\cdot\text{mg}^{-1}$ ). The Elovich model does not provide a particular mechanism insight, but it evaluates the surface heterogeneity of the adsorbents [129]. The Elovich model has been applied for the removal of aqueous pollutants by PANI-based adsorbents and the reported regression coefficients (0.85-0.95) was lower than those from the PSO and PFO models [55, 129, 188, 189].

Overall, aqueous pollutants removal by PANI-based adsorbents with nitrogen-rich functional amine and imine groups is mainly based on monolayer chemisorption. The system can be heterogeneous or homogeneous due to the morphological nature and distribution of the functional groups. However, physisorption may also take place owing to the porous structure of the polymeric adsorbent, which can contribute to the adsorption process. Moreover, diffusion may occur and act as the rate-controlling step when the system is not well agitated or the concentration of the adsorbate decreases to very low levels at the end of the adsorption.

Regeneration of the exhausted adsorbent is often investigated via adsorption-desorption reactions [165, 190, 191]. Regeneration of the adsorbent (i.e. desorption) occurs in a system that is in the equilibrium between bulk

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phase and the adsorbed phase. Desorption studies evaluate the nature of adsorption and regeneration capacity of the adsorbent [192]. Adsorption of pollutants by PANI-based adsorbents is highly affected by the solution pH. Therefore, selection of the desorption solution depends on the pH for adsorption [76, 165, 190-192]. Mashkooor and Nasar studied the adsorption of crystal violet dye from water by PANI/tectona grandis sawdust and investigated different desorption agents, such as HCl, CH<sub>3</sub>COOH, NaCl, NaOH, and H<sub>2</sub>O [189]. They reported that HCl was the best desorption agent since the maximum adsorption was observed at a base condition with pH 12. On the contrary, Ishtiaq et. al. reported effective desorption of PANI/peanut husk with NaOH as the regeneration agent, as the adsorbent showed high adsorption efficiency in acidic media [165]. To conclude, regeneration of PANI-based adsorbents can be achieved effectively by altering the solution pH due to the doping/dedoping feature of PANI, and it helps reduce the usage of adsorbents [68, 76, 189, 192].

## **5. Concluding remarks and perspectives**

This work gives a comprehensive review of PANI-based adsorbents for removal of various pollutants from drinking water and wastewater. These adsorbents have unique electrical characteristics and many other advantages, such as facile synthesis, low cost of monomers, excellent environmental stability, facile control of reversible properties by charge exchange doping and protonation, simple acid-base doping/dedoping process, reactive NH- groups, and tunable properties. In particular, doping/dedoping of PANI-based adsorbents alters the morphology, pore size, surface charge and functional groups of the adsorbents, leading to enhanced adsorbent-adsorbate interactions and thus pollutant removal performance.

PANI-based adsorbents have been used for the removal of three key types of pollutants, including heavy metals and metalloids, organic dyes, and emerging pollutants. The key adsorption mechanisms include electrostatic interactions, van der Waals forces,  $\pi$ - $\pi$  interactions, and/or hydrogen bonding. These adsorbents possess



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diverse morphologies, such as nanorod, nanotube, nanofiber, nanoflake, nanopowder depending on the synthesis conditions. Therefore, adjusting the desirable morphology with optimal properties, such as chelation, complexation, and effective surface area is required for a specific adsorbate. A size selectivity mechanism can also be achieved by adjusting the pore size of the composite adsorbent. Regeneration of PANI-based adsorbents can be effectively achieved by altering the solution pH due to the doping/dedoping feature of PANI.

In the future, engineering PANI-based sorbents for removal of emerging micropollutants can be of high interest to both academia and wider water sector. An array of techniques could be proposed to further enhance the removal capacity and selectivity in the adsorption process. For instance, incorporation of 2D or 3D high aspect ratio fillers, such as clays or graphene into PANI could further increase the effective surface area of the adsorbent. Degradation of the adsorbates, which facilitates the regeneration of the adsorbents, can be realized by incorporating catalytic fillers, such as TiO<sub>2</sub> and reactive carbon-based materials [193]. To this end, reactive carbon-based materials are of special significance due to their low costs and environmentally benign nature. Carbon materials (e.g. graphene) can be either doped with hetero-atom elements or engineered to have rich radicals and defects, which make them reactive to degrade various pollutants [194, 195]. PANI-based adsorbents can also be combined with membrane technology to engineer adsorptive membranes for various aqueous pollutants removal, which would take the advantages of the beneficial properties of each technology [193, 196].

In addition, enhancement of charge-selective adsorption could be achieved by manipulating the functional groups of the adsorbent and the introduction of specific functional groups. Furthermore, enhancement of size-selective adsorption can be informed by advanced characterization methods and achieved by manipulation of adsorbent morphology. With these possible avenues delineated, one key barrier to commercialization depends on the economic and efficient scale-up of PANI-based sorbents during manufacturing.

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