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Review

Wastewater Treatment Methodologies, Review Article

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Abstract

The problem of fresh water deficiency is of worldwide impact. The increased population in addition to various human activities has led to a drastic and continuous shortage of freshwater resources. This dilemma led to motivate the scientists to find radical and cheap solutions. One of the solutions proposed to solve this problem is to treat industrial wastewater to be suitable for agriculture by removing toxic pollutants from it. This review sheds light on the traditional and modern methods applied for this purpose with a focus on the use of natural materials as sustainable and environmentally friendly sources for creating new materials used in this regard. Furthermore, the review concentrates on the utilization of polymer nanocomposites (specially derived from or based on natural polymer) in water treatment as a relatively modest trend.

Keywords: Wastewater; Adsorption; Biological Treatment; Nanoparticles Contents

Introduction

The Problem of Water Deficiency

The shortage of water resulting from economic and population growth is considered as one of the most important fear for humankind and a limitation for sustainable development. There are very limited options to confront the challenges of fresh water shortage problem; these options include [1,2,3,4,5]:

- Desalination
- Water conservation efforts
- Recycling and reuse of process and wastewater
- Rainfall infrastructure projects (dams, reservoirs, and water carriers)
- Underground infrastructure projects

Historically, policy makers have tried to solve water scarcity problems through dam building, groundwater recharge, cloud seeding, desalination, wastewater reuse, and developing massive water transfer projects [6,7,8]. Water use is not just governed by population growth, however. In the 20th century, the world population tripled but water use increased six-fold [9,10]. In addition to the well-known water pollutants, water contamination is caused by many other factors, Figure 1.

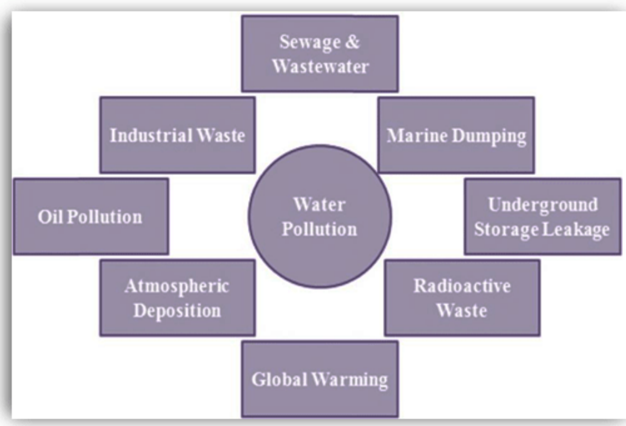

Figure 1. Sources of water contamination

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Types of Water Pollutants

Wastewater is categorized and defined according to its origin [11]. Generally, it involves two major categories:

1. The term "domestic wastewater" refers to flows discharged principally from residential sources generated by such activities as food preparation, laundry, cleaning and personal hygiene.

2. Industrial/commercial wastewater is flow generated and discharged from manufacturing and commercial activities such as printing, food, pharmaceuticals and beverage processing and production [12,13,14].

There are numerous chemical species present in water with concentrations vary from a few mg/l to a few g/l. They are referred as micro-pollutants, which even in small quantities are dangerous to man's health [15,16]. Types of water pollutants are illustrated in figure 2. Moreover, the importance of some of these contaminants is summarized in Table 1.

Traditional Wastewater Treatment Processes

The treatment of wastewater is talented by four fundamental techniques; physical, mechanical, biological and chemical [17,18,19]. They are summarized in Figure 3.

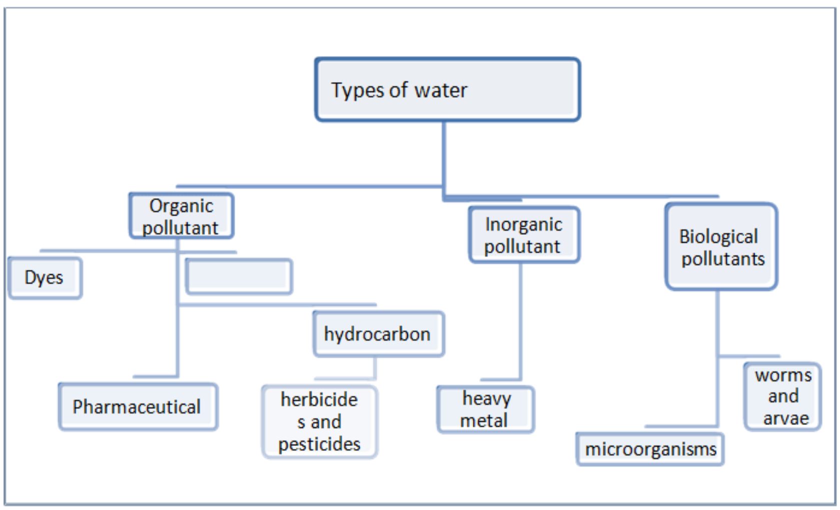

Figure 2. The most important water contaminants

Table 1: The most important contaminants and the reasons of their importance

Contaminant	Reason for importance
Pathogenic organisms	Found in wastewater can cause infectious diseases.
Suspended solids(s)	They lead to development of sludge deposits and anaerobic conditions when untreated waste water is discharged to the aquatic environment
Refractory organics	They tend to resist conventional wastewater treatment include surfactants, phenols and agricultural pesticides.
Heavy metals	They are highly toxic and environmentally hazardous species
Priority pollutants	Including organic and inorganic compounds, may be highly
Dissolved inorganic constituent	Such as calcium, sodium and sulfate are often initially add to domestic water supplies and may have to be removed for wastewater reuse.

Wastewater Treatment Methods

Physical Methods

They engage all the physical forces that applied to remove contaminants. They still figure the basis of most process flow systems used for wastewater treatment [20,21,22,23,24].

Flow equalization: This technique is applied to improve the effectiveness of secondary and basic wastewater treatment processes by leveling out operation parameters such as flow, pollutant levels, and temperature over a period [25].

Sedimentation: Sedimentation, a fundamental and widely used unit operation in wastewater treatment, involves the gravitational settling of heavy particles suspended in a mixture. This process is used for the removal of grit, particulate matter in the primary settling basin, biological flock in the activated sludge -settling basin, and chemical flow when the chemical coagulation process is used [26,27].

Flotation: In this method, air bubbles are introduced to expel solid or liquid particles from a liquid. The gas bubbles either hold on the liquid or are trapped in the particle structure of the suspended solids, enhancing the buoyant force of the combined particle and gas bubbles [28,29,30,31]. Some environmental applications of flotation are given in Table 2.

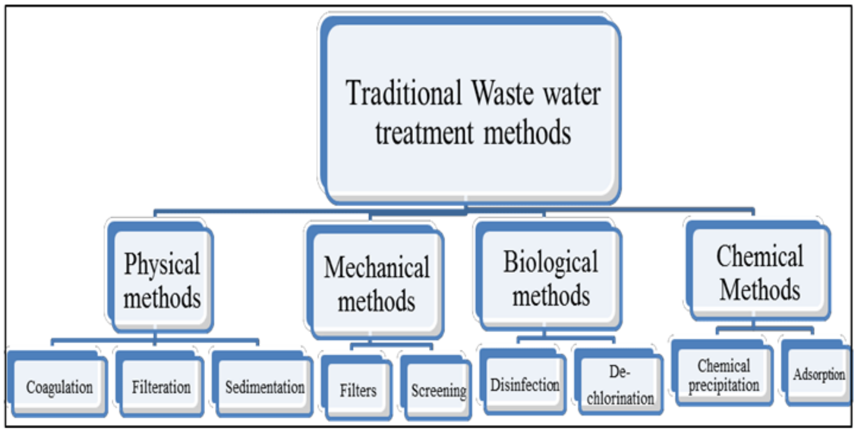

Figure 3. Traditional Waste Water Treatment Methods

Table 2: Some environmental and industrial applications of flotation [29,30,32–37]

1	Treatment of effluent with heavy metal and anions.
2	Water recycle: Anions and calcium ion removal.
3	Treatment of acid mine drainage and water reuse.
4	Treatment of soil
Industrial applications:	
1	Treatment of water for industrial and domestic use.
2	Treatment of sewage
3	Removal of microorganisms.

Mechanical Methods

Screening: This method is one of the oldest treatment methods. It removes gross pollutants from the waste stream to protect downstream equipment from damage, evade interference with plant operations and prevent obnoxious suspended materials from entering the primary settling tanks [32,33,34,35,36,37].

Filters: The filters can be classified as biological methods rather than mechanical methods. It can be considered the most commonly encountered aerobic attached-growth biological treatment process used for the removal of organic matter from wastewater.

Biological Methods

Biological unit processes are utilized to alter the finely divided and dissolved organic matter in wastewater into flocculent organic and inorganic solids [38,39,40,41,42]. In these processes, micro-organisms, particularly bacteria, adapt the colloidal and dissolved carbonaceous organic matter into a mixture of gases which is then removed in sedimentation tanks. It includes the following operations:

Disinfection

It can be defined as any process aims to destroy or prevent the growth of microbes. It intends to inactivate the microbes by physical, chemical, or biological processes. This inactivation is achieved by altering or destroying essential structures or functions within the microbe [43]. The most commonly used ways of disinfection include the following:

1. Physical agents such as heat and light;
2. Mechanical means such as screening, sedimentation, and filtration.
3. Radiation, mainly gamma rays;
4. Chemical agents.

Dechlorination

Dechlorination is the subtraction of free and total combined chlorine residue from chlorinated wastewater effluent before its reuse or discharge to receiving waters. Dechlorination is carried out by the use of activated carbon, or by the addition of a reducing agent such as sulfur dioxide (SO_2), sodium sulfite (Na_2SO_3) or sodium metabisulfite ($Na_2S_2O_5$). It is important to note that dechlorination will not eliminate toxic by-products that have already been produced.

Chemical Methods

This section discusses the main chemical unit processes, including chemical precipitation, adsorption, and other applications [44,45,46,47,48].

Chemical Precipitation

The precipitation of heavy metals takes place by reaction with certain of chemicals to form insoluble precipitates. The resultant precipitates can be removed from the water by sedimentation or filtration. The treated water is then decanted and appropriately discharged or reused. Among the chemical coagulants that are commonly used in waste-water treatment: alum ($Al_2(SO_4)_3\ H_2O$), ferric chloride ($FeCl_3.6H_2O$), ferric sulfate ($Fe_2(SO_4)_3$), ferrous sulfate ($FeSO_4.7H_2O$) and lime ($Ca(OH)_2$) [49,50].

Adsorption with Activated Carbon

Adsorption is the process of collecting soluble substances within a solution on a proper surface [51,52,53]. In wastewater treatment, adsorption with activated carbon a solid interface usually follows normal biological treatment, and is aimed at removing a portion of the remaining dissolved organic matter.

Advanced Techniques for Wastewater Treatment

Recently, researchers developed numerous techniques for achieving maximum efficiency and minimum cost. They include membrane filtration, ion-exchange, electrolysis, adsorption, etc.

Membrane Filtration

The most common membrane processes used to remove metals from the wastewater are the following: Ultra- filtration, reverse osmosis, nanofiltration, and electro-dialysis.

Ultrafiltration

Ultrafiltration (UF), Figure 4 is a membrane technique working at low trans membrane pressures for the removal of dissolved and colloidal material [54,55]. The use of ultra filtration technology for wastewater applications is a relatively recent concept. Although in the beginning, it is already commonly used in many industrial applications such as food or pharmaceutical industries. Since the membrane pore sizes are larger than dissolved metal ions in the form of hydrated ions or as low molecular weight complexes, these ions can pass easily through. To obtain high removal efficiency of metal ions, the micellar enhanced ultrafiltration (MEUF) and polymer enhanced ultrafiltration (PEUF) was proposed [56].

Micellar enhanced ultra filtration (MEUF) process has been used for the removal of copper, chromate, zinc, nickel, cadmium, serinium, arsenate, and organics like phenol, o-cresol [57,58,59,60].

Metals removal was enhanced by combining the MEUF treatment with electrolysis or with powdered activated carbon (PAC) [61,62,63]. Cetylperidinium Chloride (CPC) and Sodium dodecyl sulphate (SDS) surfactants removal from the MEUF was also enhanced by the MEUF-ACF (activated carbon fibre) combined treatment [64]. Surfactant has been recovered from the MEUF retentate solution by treating the retentate with HNO_3, $H2SO_4$, HCl, NaOH solution but retentate solution needs further treatment [65]. Electrolysis was found better in the separation of metal and surfactant from the MEUF retentate solution [66,67,68]. The main parameters affecting PEUF are metal and polymer type, the ratio of metal to polymer, pH and existence of other metal ions in the solution [69,70,71].

Reverse Osmosis

For the last few years, a great attention has been paid on the development of unconventional methods for wastewater treatment, such as pressure driven membrane operations, namely ultra filtration which helps eliminate colloids, suspended and macromolecular matter, and reverse osmosis, which helps remove mineral substances and low-molecular organic compounds [72].

The reverse osmosis (RO) process uses a semi-permeable membrane, allowing the liquid that is being purified to pass through it, while rejecting the contaminants. RO is one of the techniques able to remove a wide range of dissolved species from water, Figure 5. It accounts for more than 20% of the world's desalination capacity [73].

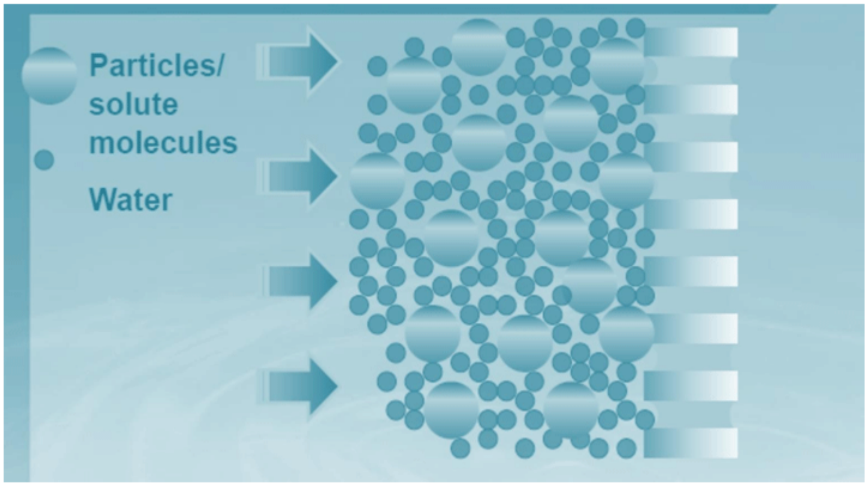

Figure 4. Pressure driven membrane separation

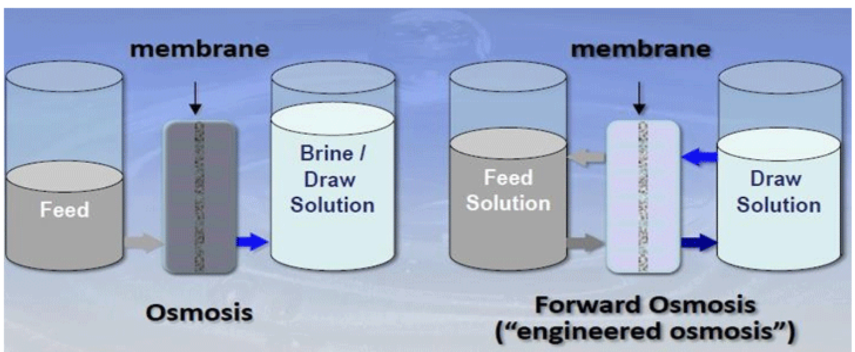

Figure 5. Osmotically driven membrane process

Nano-Filtration

The application of membrane separation methodology in water and wastewater treatment is growing due to stringent water quality standards. Nanofiltration (NF) is one of the widely used membrane processes for water and wastewater treatment in addition to other applications such as desalination. NF has replaced reverse osmosis (RO) membranes in many applications due to lower energy consumption and higher flux rates [74,75,76].

Nano-filtration (NF) is the intermediate process between UF and RO. NF is an attractive technology for the elimination of heavy metal ions such as nickel, chromium, copper and arsenic from wastewater [77,78,79]. There are many reports on the removal of heavy metal by NF and RO membrane [80,81,82]. Mikulasek and Cuhorka [83] studied the performance of different NF and RO membranes in removing toxic lead ions from wastewater. The influence of the operational variables, i.e. the pressure applied, feed solution pH, and feed solution concentration, on the ability of the NF membranes to remove ions was evaluated.

Electro dialysis

Electro dialysis (ED)- Figure 6- is one of the first commercially available large scale water desalination processes based on membranes and is still widely used all over the world. In ED electric potential is used as a driving force and ion exchange membrane is applied between anode and cathode [84,85]. The membrane is defined as a selective barrier between two phases and it can be formed from natural and synthetic material including organic and inorganic polymer, ceramic and metal material. The membranes are essentially of two main types: cation- exchange and anion-exchange membranes [86]. This process has been widely used for the production of drinking and process water from brackish water and seawater, treatment of industrial effluents, recovery of useful materials from effluents and salt production [87]. Removal of copper ions by the application of ED system was study by Caprarescu et al. [88]. In order to achieve copper ions removal two different ion exchange membranes were used.

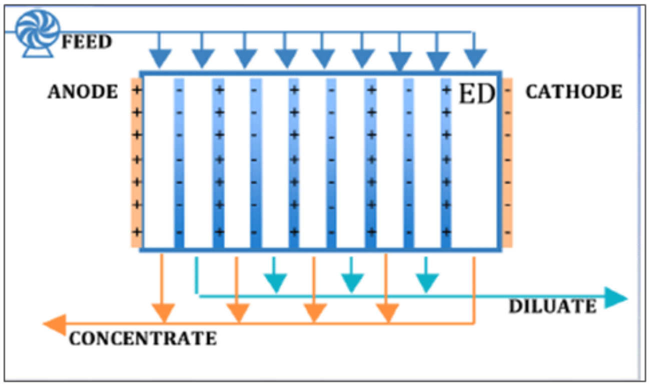

Figure 6. Electrodialysis and reverse electrodialysis

Adsorption

Adsorption is known to be one of the greatest of the technologies applied for the wastewater treatment because it is an effective, economical and ecofriendly treatment technique. It is strong enough to realize water reuse obligation and high runoff standards in the industries. Adsorption is basically a mass transfer process by which the metal ion is transferred from the solution to the surface of sorbent, and becomes bound by physical and/or chemical interactions [89,90]. The functional groups thus play an important role in determining the effectiveness, capacity, selectivity, and reusability of these adsorbents.

In general, the main steps involved in adsorption of pollutants on solid adsorbent are:

1. Transfer of the metal ion from bulk solution to the outer surface of the adsorbent.
2. Internal mass transfer by pore diffusion from outer surface of adsorbent to the inner surface of porous structure.

3. Adsorption of adsorbate onto the active sites of the pores of adsorbent.

4. The overall rate of adsorption is governed by either film formation or intra particle diffusion or both as the last step of adsorption are very fast as compared to the other two steps.

A comparison between the advantages and disadvantages of the above-mentioned methods is given in table 3.

Types of Sorbents

Sorption is a physical and chemical process by which one substance becomes attached to another. This type of attachment lies under the following, Figure 7:

1. Absorption – "the incorporation of a substance in one state into another of a different state" (e.g., liquids being absorbed by a solid or gases being absorbed by a liquid);

2. Adsorption – the physical adherence or bonding of ions and molecules onto the surface of another phase (e.g., reagents adsorbed to a solid catalyst surface);

3. Ion exchange – an exchange of ions between two electrolytes or between an electrolyte solution and a complex.

4. Commonly, in physical adsorption, the attractive forces between adsorbed molecules and the solid surface are Van der-Waals forces of attraction and reversible adsorption (Desorption) occurs when they are weak in nature.

5. (Ad) sorption at a solid–liquid interface is a complex process playing a critical role in several industrial applications as well as in the fate and passage of chemical pollutants in the environment. In industry, the sorption techniques concerning solid sorbents are widely used to remove certain classes of chemical pollutants from waters, especially those that hard and resistant [90].

Recently, ion exchange or chelating fiber as one type of adsorbents has been widely used. In this regard, Rafati et al. [44] studied the removal of the chromium (VI) ion from aqueous solutions with the Lewatit FO36 ion-exchange resin at different conditions. The effects of adsorbent dose, initial metal concentration, contact time, and pH on the removal of chromium (VI) were investigated. The batch ion exchange process was relatively fast and it reached equilibrium after about 90 min of contact. The ion exchange process, which is pH dependent showed maximum removal of chromium (VI) in the pH range 5.0-8.0 for an initial chromium (VI) concentration of 0.5 mg/dm^3.

Table 3: Advantages and disadvantages of different treatment methodologies

Treatment technique	Advantages	Disadvantages
Electrochemical oxidation	Does not require chemicals, high pressures, or high temperatures.	Low selectivity and low reaction rates
Biological process	Ecologically favorable process.	High capital and operational cost. Handling and disposing the secondary post problems.
Adsorption	Cost- effective method. Easy availability and operation. Most profitable process and more efficient than the conventional methods	Merely removes the pollutants from one phase (aqueous) to another (solid matrix). Expensive process for regeneration especially if the pollutants are strongly bond to the adsorbents.
Advanced oxidation processes(AOP) **(i) Ozonation** **ii) UV** **(iii) UV/H_2O_2** **(iv) O2/UV/H_2O_2** **(v) Fenton reaction**	Powerful oxidation technique oxidizes a large number of organic and inorganic materials An effective method that typically does not leave any byproduct which are harmful to the environment An effective technique in the oxidation and mineralization of most organic pollutants. Ease of formation of .OH radicals. Most effective process due to the fast generation of. OH radicals. Can treat a wide variety of contaminants. Simple process. Easy availability of chemicals.	More complex technology and requires high capital/ operational cost. High electric consumption. Less effective if, the waste water has amounts of particulates which can absorb UV light. Less effective, when the wastewater has high absorbance. High operational cost. Needs to compare with high turbidity, solid particles, and heavy metal ions in the aqueous stream. High operational cost. Production of sludge iron waste and handling the waste pose logistical problems
(vi) Photo- Fenton reaction **(vii) Heterogeneous photocatalysis**	Reduction of sludge iron waste compared to original Fenton reaction. Effective and fast degradation. Long- term stability at high temperature. Resistance to attrition. Low- cost and environmentally begins treatment technology.	Needs a controlled PH medium for better performance. Could form byproducts that can be harmful to the environment. Requires efficient catalysts that can absorb in the visible region.

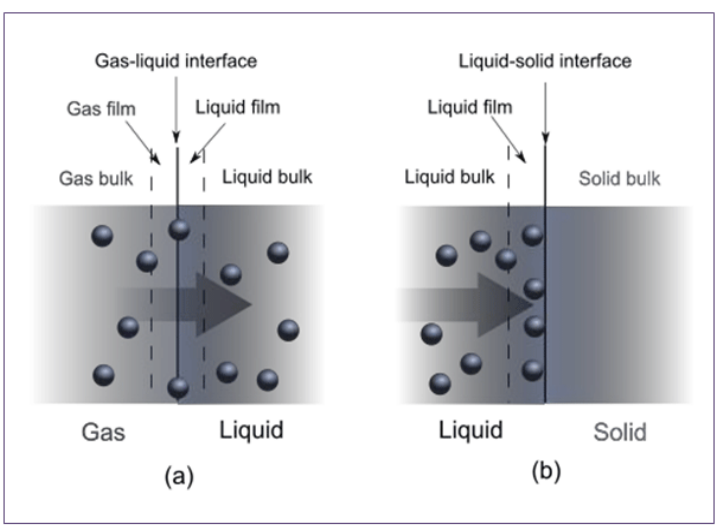

Figure 7. Gas–liquid absorption (a) and liquid–solid adsorption (b) mechanism.

Hydrogels

Recently, hydrogels have attracted great concern since they can be applied as selected adsorbents for removing heavy metal ions from wastewater. This is mainly due to the three-dimensional network structure and their ability of incorporating different functional groups into the polymeric networks [91,92,93,94,95]. Hydrogels can absorb large amounts of water based on intrinsic properties such as polarity, degree of crosslinking between the network chains, chain flexibility, and free volume. The water holding capacity of hydrogels also depends on the external stimuli such as pH, temperature, and ionic strength. Thus, Significant needs and efforts have been made in the development of novel hydrogels for early detecting and effectively removing heavy metal ions from environments [96,97,98,99].

However, there are two main issues for practical applications of hydrogels, one of which is to improve the mechanical strength of the hydrogel; another is to access functional groups into the hydrogel structure. To solve the first problem, hydrophobic component can be added into the structure [100]. Hydroxy ethyl methacrylate (HEA) is a hydrophilic monomer that has been studied widely as the backbone of copolymers [101].

Abdel-Aal [101] prepared two copolymer hydrogels of maleic acid (MA) and 2-hydroxyethylacrylate (HEA) or acrylamide (AAm) for removal of some organic dyes. It was also reported that the absence of HEA increased the cross linked network structure, and HEA/MA hydrogel showed higher adsorption capacity than AAm/MA hydrogel. The synthesis of copolymeric hydrogels containing various functional groups, especially imino (INH2) or carboxylic (ACOOH) groups, has received growing concern. In this regard, Iminodiacetic acid functionality has been introduced on styrene–divinyl benzene co-polymeric beads and characterized by FT-IR in order to develop weak acid based cation exchange resin [102]. This resin was evaluated for the removal of different heavy metal ions namely Cd(II), Cr(VI), Ni(II) and Pb(II) from their aqueous solutions. The results showed better attraction of resin towards Cr(VI) for which 99.7% removal obtained in optimal conditions. The order of removal is as following Ni(II) > Pb(II) > Cd(II) with 65%, 59% and 28% respectively. Thorough studies of Cr(VI) removal has been carried out to investigate the effect of pH, resin dose and metal ion concentration on adsorption concluded that complexation enhanced the chromium removal efficacy of resin significantly, which is strongly pH dependent. The findings were also supported by the comparison of FT-IR spectra of neat resin with the chromium- adsorbed resin.

Maria et al. [103] prepared two chelating resins bearing iminodiacetate (IDA) groups derived from acrylonitrile- divinylbenzene (AN-DVB) copolymers, Figure 8. These resins were tested as sorbents for heavy metal ions like: Pb(II), Cd(II) and Zn(II) from aqueous solutions by batch and column techniques. The overall adsorption tendency of CRs toward Pb(II), Cd(II) and Zn(II), under non-competitive conditions showed the following order: Cd(II)> Pb(II)> Zn(II). Selectivity studies were performed in ternary mixture of Pb(II), Cd(II) and Zn(II) to verify the selective separation of the synthesized resins towards heavy metal cations. The results revealed that the resins with IDA groups showed high selectivity toward Pb(II), both in batch and column techniques. Regeneration of the resins was achieved using 0.1 M HCl solution.

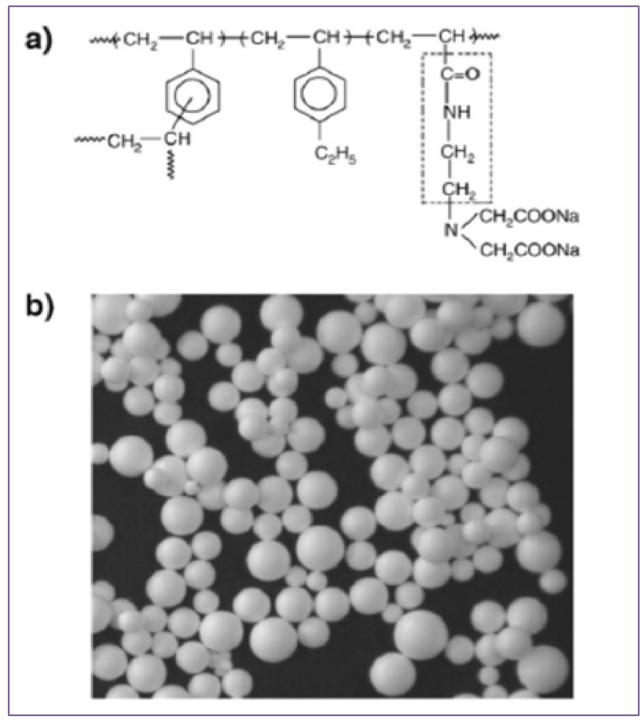

Figure 8. (a) and (b): The structure of investigated CRs and an image taken from the optical microscope for CRs

Hydrogel Nanoparticles

Recently, utilization of polymer nanocomposites - especially in the form of hydrogel- for the removal of heavy metal ions from the contaminated waste water has been established [104,105,106,107]. These nanocomposite hydrogels could swell in water so that a high adsorption capacity might be achieved [108].

Hydrogels composed of acrylic, vinylic, and other functional monomers, such as acrylic acid (AA), acryl amide (AM), 2-acrylamido2-methyl-1-propane sulfonic acid (AMPS), hydroxyl ethyl methacrylamide (HEMA), N- isopropyl acrylamide (NIPAM), N-vinyl imidazole (NI), and 4-vinyl pyridine (NVP), have been established to be excellent adsorbents for heavy metals and some other soluble species [109,110].

Mahida and Manish [110] investigated the synthesis of superabsorbent poly (NIPAAm/DAPB/ AA) amphoteric nanohydrogels by inverse microemulsion polymerization. The prepared hydrogels were applied for the removal of heavy metal ions such as Pb(II) and Hg(II) from aqueous solution. The difference between wet and dry nanohydrogels was characterized by FT-IR, TGA, SEM, and EDX analyses. The particle size of hydrogel nanoparticles was measured from TEM micrograph to be between 30 and 40 nm [111, 112,113,114,115,116]. Specific surface area and pore volume of nanohydrogel were investigated with BET and BJH analysis. They also studied the factors affecting maximum removal efficiency of Pb(II) and Hg(II) metal ions, i.e. treatment time, initial metal ion concentrations, pH values, and adsorbent dose [117,119,120,121,122,123]. From the adsorption studies, the maximum removal efficiency of metal ions toward the nanohydrogel was found to be in the following order: Pb(II) > Hg(II).

Furthermore, the adsorption of Cu(II) ions from aqueous solutions onto poly(acrylic acid-co-acrylamide) hydrogels was investigated [124,125,126,127,128,129,130]. The hydrogels were prepared via free-radical solution polymerization and the maximum metal uptake was verified by varying the ratio of acrylamide/acrylic acid moieties on the surfaces of hydrogels and the amount of cross-linking agent [131,132,133,134,135,136,137,138,139]. Swelling results indicated that, by appropriate selection of cross-linking agent amount and monomer ratio, hydrogels can be swollen up to 70,000%.

Low Cost Adsorbents

Most sorbents used for the removal of heavy metals from wastewater are derived from either modified natural polymers or agricultural waste materials [140,141,142143,144,145]. These materials are abundant, environmentally friendly, reusable and low cost, Table 4.

Table 4: Some natural polymers and agricultural wastes for heavy metal removal

Natural polymers	Metal ion removed	Ref.	Agricultural wastes	Metal ion removed	Ref.
Starch	Ni, Cu, Pb Zn, Pb, Cu, Ni, Fe and Cd Pb, Cu Ni, Zn, Cd, Pb	[121] [122] [123] [124]	Saw dust	Cr, Pb	[137]
Cellulose	Pb , Cd , Zn, Cu , Ni and Co	[125] [126]	Orange peels	Cr Cd, Zn, Cr Cd, Cu, Pb Ni Cu	[138] [139] [140] [141] [142]
Chitosan	Pb Cd, Co, Pb and Zn As, Zn Cd	[127] [128] [129] [130]	Potato peels	Cu Pb, Cd, Zn Pb Cu	[143] [144] [145] [146]
Rosin	Cr	[131]	Maize cob and husks	Zn (II), Cd (II) and Pb (II) Pb (II)	[147] [148]
Guar gum	Cr Cd Cu	[132], [133], [134], [135] [136] [137]	Rice husk and rice straw	Pb, Cd, Cu, and Zn Cd Cr, Pb, Cu Cr Pb, Cr, Cu Cr and Ni	[149] [150], [151] [152] [153] [154] [155]

Other materials were commonly utilized. For instance, Zahir et al [146]. used papaya seed, egg shell, and coconut leaf powder as adsorbent for the removal of heavy metals such as lead, cadmium and chromium from wastewater. They found that the three adsorbent removed some heavy metals effectively. Chicken egg Shell was efficient in removing 85, 82, and 86 % of chromium, lead, and cadmium respectively. Coconut leaf powder used for removing 87, 90 and 85 % of chromium, lead and cadmium respectively. Papaya seed powder is used for removing 80, 85 and 79 % Chromium, Lead and Cadmium respectively from wastewater from the initial metal ion concentration of 100 ppm solution. Furthermore, agricultural waste by-products such as fly ash were used in a case study for the treatment of the EL-AHLIA Company wastewater for electroplating industries. Results showed that low cost adsorbents effectively removed Fe, Pb, Cd, Cu and Ni with a concentration range of 20–60 mg/l [147].

Furthermore, Cyclodextrine (Figure 9) was modified chemically and applied for removal of some heavy metals due to its unique properties. In this regard, carboxy methyl β cyclodextrin, poly(ethylene glycol) β cyclodextrin and their magnetic counterparts were prepared and applied in elimination of copper and lead from simulated solution, The scheme of chemical modification of β cyclodextrin is illustrated in figure 10. The effect of some factors such as pH, concentration of the metal ion, temperature and contact time on removal effectiveness have been studied [148].

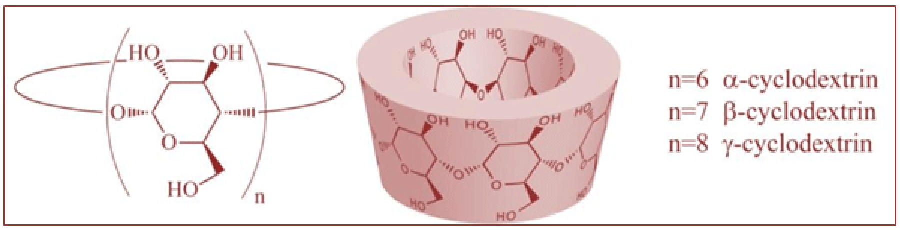

Figure 9. Chemical structure of native cyclodextrines

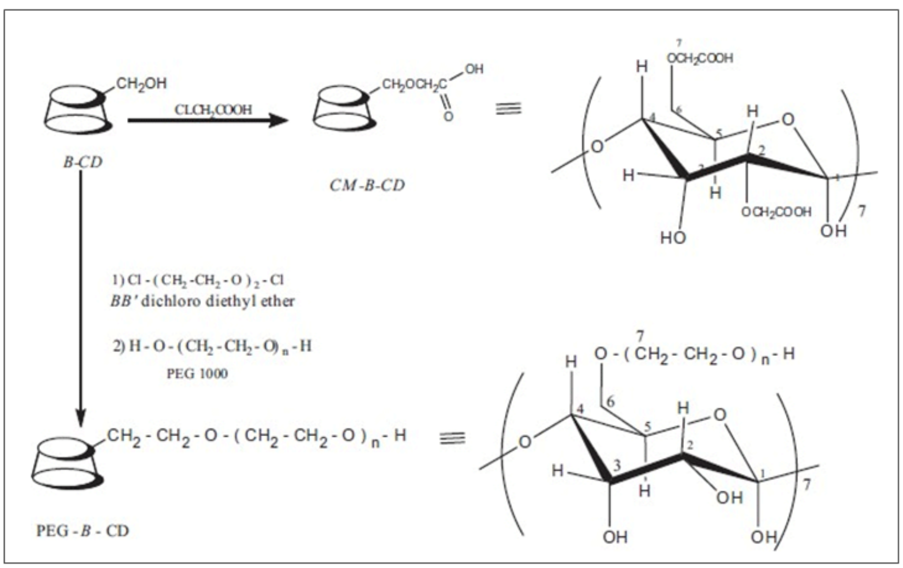

Figure 10. Modification reactions of β -CD.

Magnetic Nanocomposites for Heavy Metal Removal

As mentioned earlier, many techniques have been applied to eliminate heavy metals from wastewater both in the past (conventional techniques) and in recent years (alternative techniques) [149]. All these methods show different drawbacks and problems; therefore, innovative methods of treatment must be developed in the near future. Iron oxide nanocomposites may be a proficient means to answer this problem.

The major improvement of nanocomposites over all the applied methods is that they can efficiently be removed, after their application, simply by applying a magnet. Furthermore, the surface of these nanoparticles can be easily fabricated by different functional groups thus combining the advantages of high surface area with numerous functionalities.

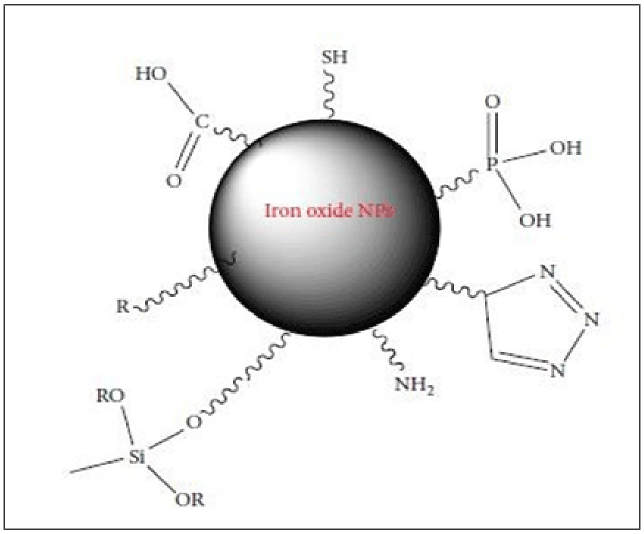

Figure 11. Surface modification of iron oxide nanoparticles [149]

This contribution provides a deep look on the elimination of heavy metals from water resources using magnetic separation. Nanocomposites based on magnetite and natural polymers and are widely known [150]. For example, carboxymethyl-cyclodextrin (CM-CD) has the ability to complex heavy metals such as cadmium, nickel, copper, and mercury through the interactions between the metal ions and –COOH functional groups. In this regard, Badruddoza et al [161]. developed a novel nano-adsorbent based on carboxymethyl β cyclodextrin fabricated with Fe_3O_4 nanoparticles, Figure 12. The prepared nanoparticles (CMCD-MNPs) were used for removal of copper ions from aqueous solution. The grafted CM-β-CD on the Fe_3O nanoparticles contributes to an enhancement of the adsorption capacity because of the strong abilities of the multiple hydroxyl and carboxyl groups in CM-β-CD to adsorb metal ions. The adsorption of Cu^{2+} onto CMCD-MNPs is found to be dependent on pH and temperature. FTIR and XPS showed that Cu^{2+} adsorption onto CMCD-MNPs mainly involves the oxygen atoms in CM-β-CD to form surface-complexes. In addition, the copper ions can be desorbed from CMCD- MNPs by citric acid solution with 96.2% desorption efficiency and the CMCD-MNPs exhibited good recyclability [151].

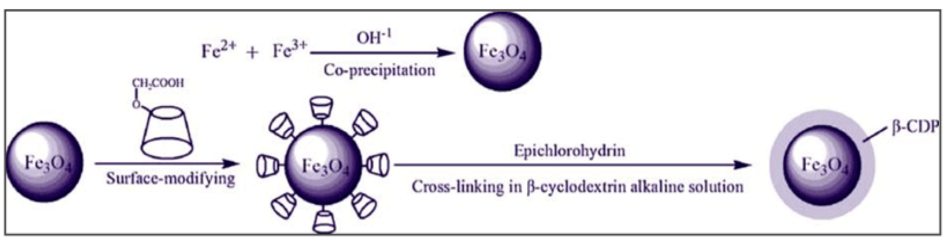

Figure 12. Illustration of preparation steps of cross-linked b-cyclodextrin polymer/Fe_3O_4 composite nanoparticles with core-shell structures.

The starch extracted from potato peels was modified with acrylic acid. Nanoparticles composed of grafted starch- acrylic acid copolymer and Fe3O4 (modified potato starch-magnetic nanoparticles, MPS-MNPs) were synthesized. The prepared Nanoadsorbents were utilized for selective abstraction of Pb2+, Cu2+, and Ni2+ ions from water [152]. The grafting reaction of acrylic acid onto starch is provided in Figure 13.

Chitosan has been intensively studied as a base material for magnetic carriers because of its significant biological and chemical properties. In general, chitosan itself does not exhibit magnetic properties; therefore, an additional magnetic component must be added in order for the particles to function optimally in particle separation. General preparation of chitosan nanoparticles is given in Figure 14. For more illustration, a proposed scheme of magnetic separation process based on using magnetic chitosan composites (MCC) is given in Figure 15. The magnetic particles that are typically embedded into chitosan include Fe_3O_4, Fe_2O_3, $NiFe_2O_4$, $CoFe_2O_4$, $CuFe_2O_4$ and $ZnFe_2O_4$ [153,154,155,156], Figure 16.

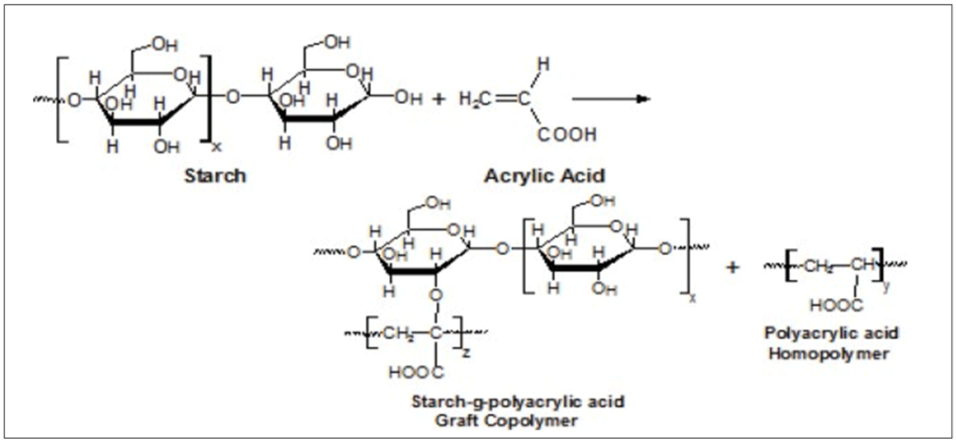

Figure 13. Synthesis of acrylic acid-starch graft copolymers

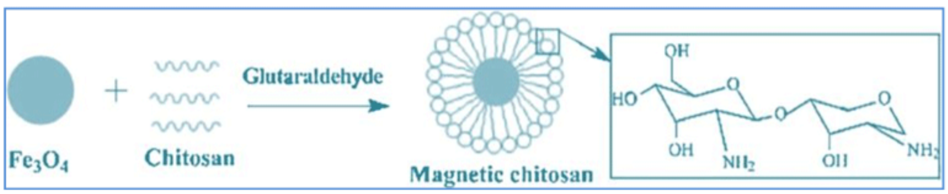

Figure 14. Schematic formation of magnetic chitosan from magnetite and chitosan

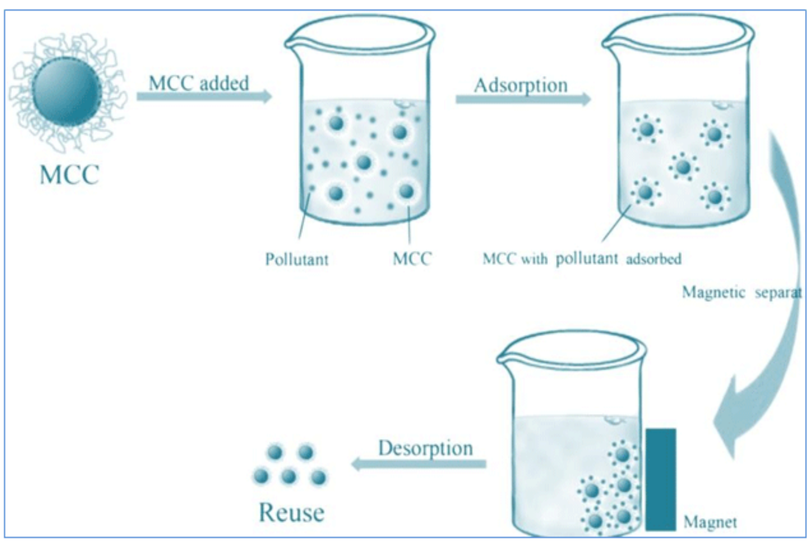

Figure 15. A schematic diagram of the magnetic separation process based on using magnetic chitosan composites (MCC).

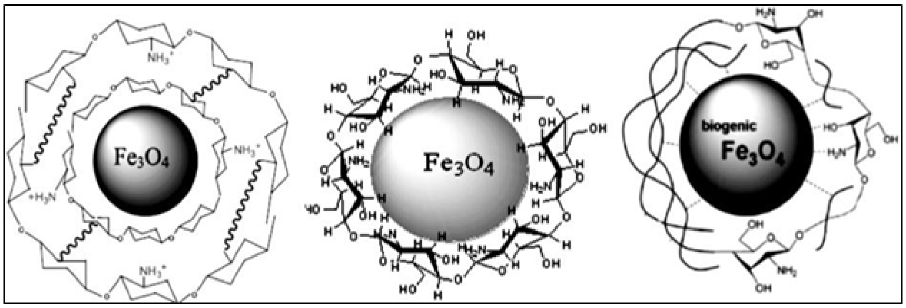

Figure 16. Different forms of chitosan bonded Fe3O4 materials

In this regard, Pineda et al [157]. prepared Chitosan-coated magnetic nanoparticles (CMNP) in one-step by precipitation in a high-aqueous phase content reverse micro emulsion. The high aqueous phase concentration led to increase the productivity of nanoparticles in the micro emulsion. The obtained nanoparticles showed a narrow particle size distribution with an average diameter of 4.5 nm. Furthermore, they present superparamagnetism and high magnetization values; close to 49 emu/g. The prepared nanoparticles showed high heavy ion removal capability; as demonstrated by their use in the treatment of Pb2+ aqueous solutions; from which lead ions were completely removed within 10 min.

Tran et al [158]. proposed a simple and effective process to prepare chitosan/magnetite nanocomposite beads with saturation magnetization value as high as uncoated Fe3O4 nanoparticles (ca. 54 emu/g). This was achieved by coating the nanoparticles with very thin chitosan layer. Chitosan layer on the magnetic Fe3O4 nanoparticles can coordinate with heavy metal ions, leading to removal of those ions with the aid of external magnets. Maximum adsorption capacities for Pb(II) and Ni(II), occurred at pH 6 and under room temperature were as high as 63.33 and 52.55 mg/g respectively, in accordance with Langmuir isotherm model. The scheme of the proposed synthesis is illustrated in Figure 17.

A simple method was introduced to prepare magnetic chitosan nanoparticles by co-precipitation via epichlorohydrin cross-linking reaction [159]. The average size of magnetic chitosan nanoparticles is estimated at ca. 30 nm. It was found that the adsorption of Cr(VI) was highly pH-dependent and its kinetics follows the pseudo-second-order model. Maximum adsorption capacity was reached at pH 3 and at room temperature. The nanoparticles were thoroughly characterized before and after Cr(VI) adsorption. From this result, it can be suggested that magnetic chitosan nanoparticles could serve as a promising adsorbent for Cr(VI) in wastewater treatment technology.

Jianbo et al [160]. could prepare nano iron oxide impregnated in chitosan bead (NIOC) by using Fe(III) salts and chitosan. These nanocomposites were utilized for aqueous Cr(VI) elimination via sol-gel technique with no additional crosslinking agent. The proposed mechanism of this process is illustrated in Figure 18. The maximal adsorption capacity (69.8 mg/g) was reached at pH 5.0 and 20^0C. Cr (VI)-loaded NIOC could be effectively regenerated by alkaline solutions.

Other polysaccharide magnetic composites are also commonly applied for treating wastewater. Cellulose nanomaterials were used in different forms for water treatment technologies [161]. Cellulose-based beads with micro and nanopore structure were fabricated via an optimal extrusion dropping technology from NaOH/urea aqueous solution [162]. The composite beads incorporated with carboxyl ornamented magnetite nanoparticles and nitric acid modified activated carbon showed highly effective removal performance for Cu^{2+}, Pb^{2+}, and Zn^{2+}. Their structure and properties were investigated. Additionally, the adsorption equilibrium, kinetics, and thermodynamics of Cu^{2+}, Pb^{2+}, and Zn^{2+} by the prepared composite adsorbents were examined. The results revealed that these adsorption processes were spontaneous endothermic reactions and determined by combination of physical and chemical adsorptive mechanism. A Schematic depiction of preparation of MCB and the adsorption mechanism of heavy metal ions by MCB is provided in Figure 19.

The development of guar gum containing magnetic nanoparticles and their application in extraction of Pb(II) from wastewater has been investigated [163]. The magnetic nanoparticles (MNPs) were prepared in-situ in poly (acrylamide)-Guar gum P(GG-AM) hydrogels. The formation of magnetic nanoparticles in the hydrogel networks was determined by Fourier transform infrared spectroscopy (FTIR), Scanning electron (SEM) microscopy studies confirmed the formation of MNPs throughout the hydrogel networks.

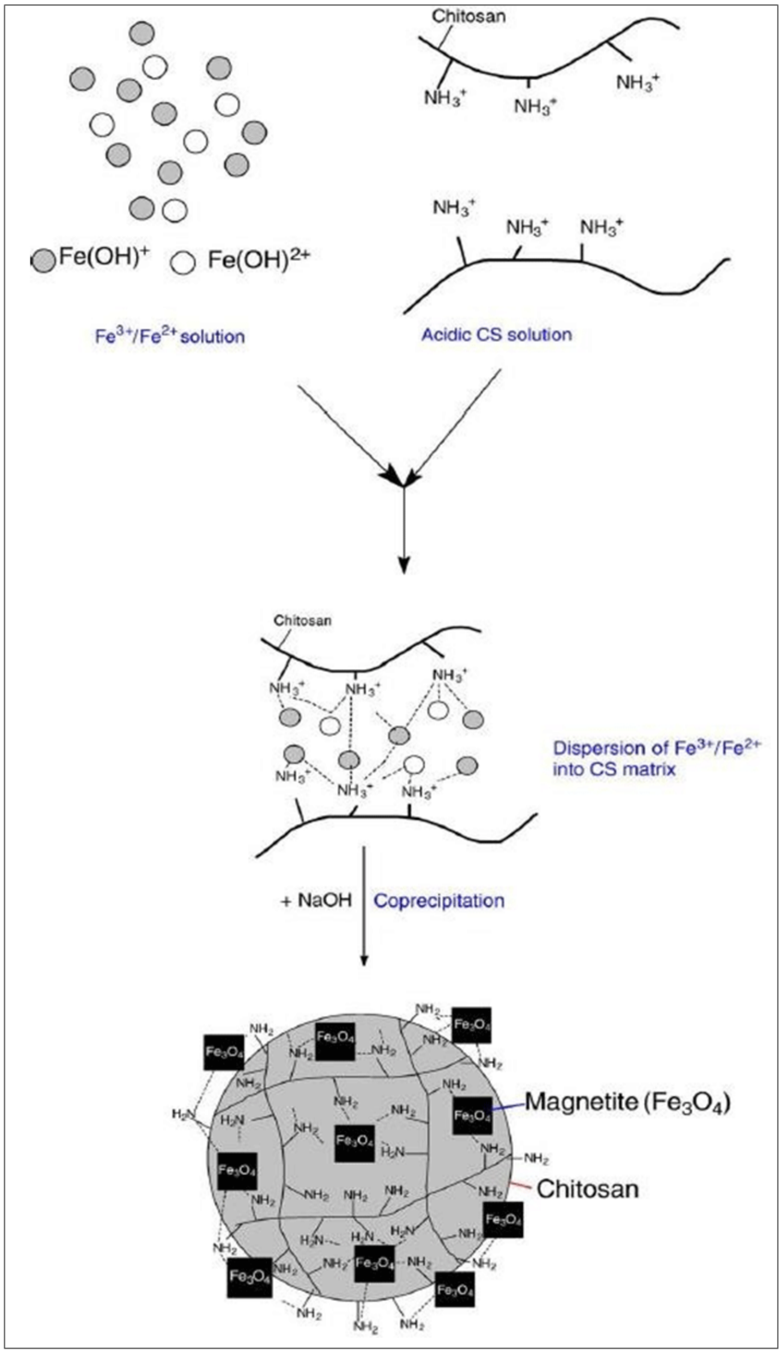

Figure 17. Schematic formation process of chitosan/magnetite composite beads [158].

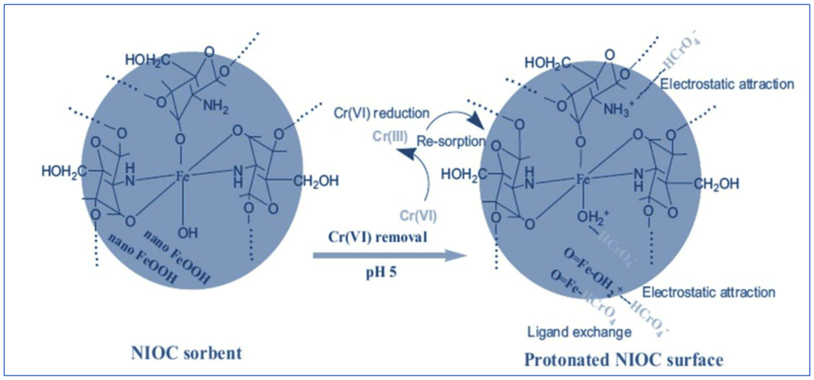

Figure 18. Schematic diagram of proposed Cr(VI) removal mechanisms on NIOC [160].

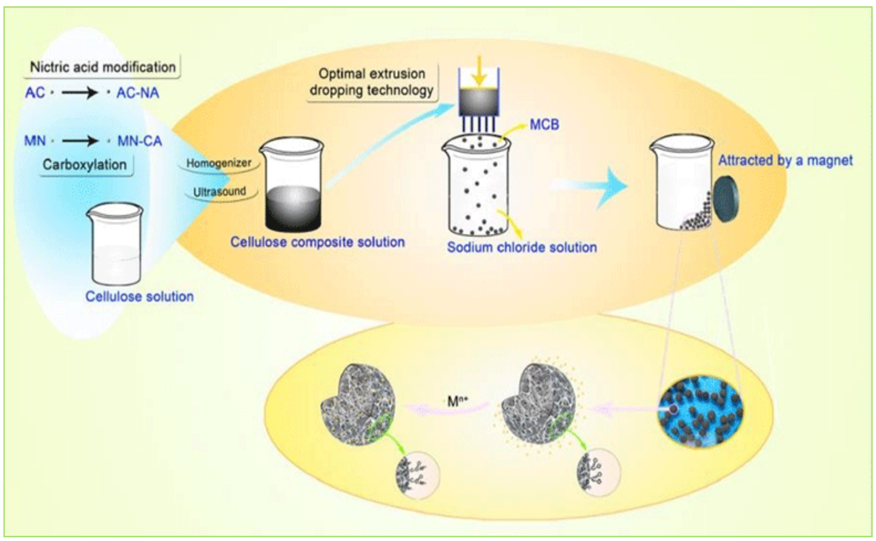

Figure 19. Preparation of MCB and the adsorption mechanism of heavy metal ions by MCB [162]

Furthermore, an efficient method for preparing Fe_3O_4- layered double –hydroxide (LDH) - guar gum bionanocomposites (GLF-BNCs) was introduced [164]. First, the LDH coated Fe_3O_4 nanoparticles were simply synthesized, using ultrasonic irradiation and then guar gum as a biopolymer were linked onto Fe_3O_4@LDH via an in situ growth method. Furthermore, the GLF-BNCs had the ability to remove cadmium ions (Cd^{2+}) from the aqueous solutions. The schematic illustration for the synthesis of LDH/GG BNCs is given in Figure 20.

Carboxymethyl cellulose magnetic nanocomposites were also utilized in elimination of heavy metals. Yawen et al. [165] synthesized acarboxymethyl cellulose (CMC)-modified Fe_3O_4 composite (denoted as Fe_3O_4@CMC) via a simple co-precipitation approach. The data obtained from FT-IR, zeta potential and thermogravimetric analysis indicated that CMC was successfully coated on the Fe_3O_4 surfaces with a high weight percent (about 30 %(w/w).

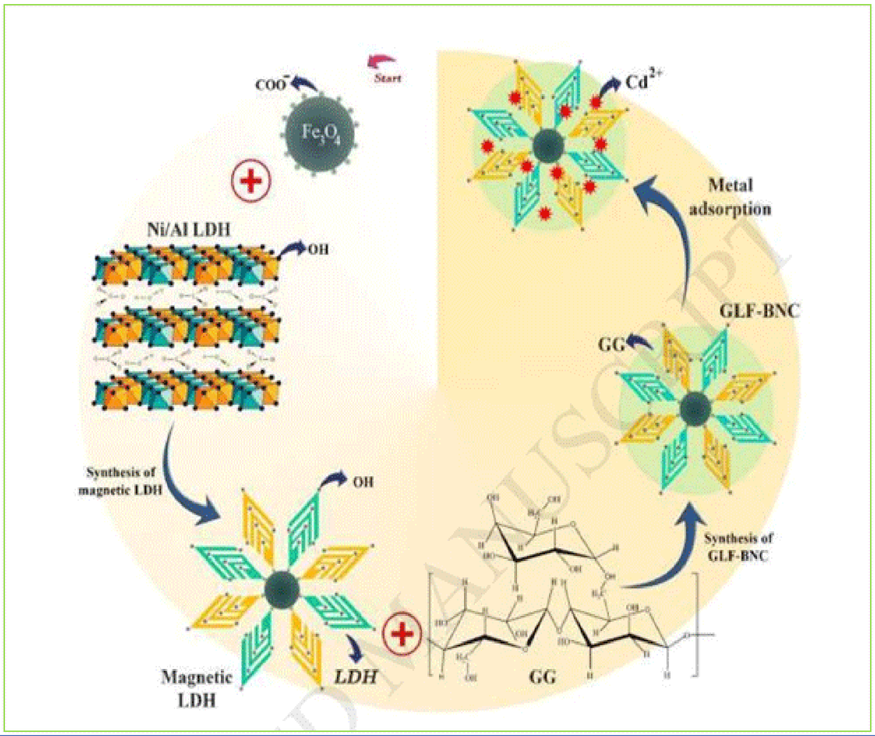

Figure 20. Synthesis of LDH/GG BNCs and Cd elimination mechanism [174].

The prepared composite was stable in acidic solution and could be easily collected with the aid of an external magnet. The ability of the Fe3O4@CMC composite to remove Eu(III) was verified as a function of various environmental parameters such as contact time, solution pH, ionic strength, solid content and temperature. The maximum sorption capacity at 293 K was calculated to be 2.78 9 10^{-4} mol/g, being higher than the series of adsorbent materials reported to date. The spectroscopic characterization revealed that Eu(III) was attached on the hydroxyl and carboxyl sites of Fe_3O_4@CMC via inner-sphere complexation. Overall, the Fe_3O_4@CMC composite could be successfully utilized as a cost-effective adsorbent for the removal of trivalent lanthanide/actinides from radioactive wastewater. For illustration, the detailed processes of synthesis and metal elimination are clarified in Figure 21.

Furthermore, preparation of nano composite hydrogels with magnetic properties was introduced, [166]. This was achieved in two steps. The first step implied preparation of semi IPN hydrogels based on sodium carboxy methyl cellulose (NaCMC)/poly(acrylamide) cross linked with N,N-methylene bisacrylamide by redox polymerization technique. The second step involved fabrication of those hydrogels onto the surface of magnetic nanoparticle. The synthesized hydrogels with magnetic properties were further utilized for the removal of toxic metal ions. The sorption behavior was investigated with respect to contact time, initial pH, and initial metal ion concentration to determine the optimum sorption conditions. The adsorption experiments of Cu and Ni on the semi IPN gels proved that the composite hydrogels have superior properties for removal of toxic metal ions compared to pure hydrogels.

Arabic gum is another natural, harmless and environment friendly polymer containing active functional groups like carboxylate and amine groups, Figure 22.

In this respect, a novel magnetic nano-adsorbent was developed by treating Fe_3O_4 nanoparticles with gum arabic to eliminate copper ions from aqueous solutions [167]. Gum arabic was attached to Fe_3O_4 through the interaction between the carboxylic groups of gum arabic and the surface hydroxyl groups of Fe_3O_4. The data revealed that both the naked magnetic nanoparticles (MNP) and gum arabic modified magnetic nanoparticles (GA-MNP) could be used for the adsorption of copper ions via the complexation with the surface hydroxyl groups of Fe_3O_4 and the complexation with the amine groups of gum arabic, respectively. The adsorption rate was so fast that the equilibrium was achieved within 2 min due to the absence of internal diffusion resistance and the adsorption capacities for both MNP and GA-MNP increased with increasing the solution pH. In addition, the copper ions could desorb from GA-MNP by using acid solution and the GA-MNP exhibited good reusability.

Future Perspectives

Due to several environmental and economic concerns, there is a rapid growth in the field of wastewater treatment. The utilization of natural polymers either neat or modified is the cornerstone in the process of water treatment.

The fabrication of iron oxide with natural polymer and preparation of iron oxide nanomaterials has received much attention due to their distinctive properties, such as extremely small size, high surface-area-to-volume ratio, surface modifiability, excellent magnetic properties, ease of recovery, recyclability, regeneration, and great biocompatibility. A number of environmental clean-up technologies have been established in wastewater treatment which applied iron oxide nanomaterials as nanosorbents and photocatalysts. Up to date, iron oxide based immobilization technologies are mainly for enhanced removal efficiency of heavy metals. Future trends will focus on applications of these fabulous materials in removal of other types of pollutants such as organic dyes, oil spill, and hydrocarbons. Few works were published. In this regard, Fernando et al [168] produced magnetic polymer composites for cleaning of oil spills on water. The polymer matrix consists of a biopolymer material (alkyd resin), which was cured with TDI in the presence of a magnetic powder (maghemite, g-Fe_2O_3), which is impregnated into the final polymer material in situ. Oil removal tests applied with polymer beads proved that the polymers can be used to remove considerable amounts of oil from the surface of aqueous environments (1:8 wt/wt polymers: oil).
Atta et al [169]. could prepare a new magnetic powder based on magnetite and amidoximes based on rosin. Amidoximes were prepared from a reaction between hydroxylamine and rosin/acrylonitrile adducts. The produced rosin amidoximes were used as entrappers for magnetite nanoparticles to prepare hydrophobic coated magnetic powders, Figure 22. The produced powder was utilized as a petroleum crude oil collector. The results show promising data for the separation of the petroleum crude oil from aqueous solution in environmental pollution cleanup. There is still a need to intensify the utilization of magnetic nanoparticles fabricated with natural polymers in water treatment application.

Moreover, special attention will be paid to elimination of pesticides from agricultural water. Definitely, the hazards of pesticide residues on human health is a worldwide problem, as human exposure to pesticides can ensue through ingestion, inhalation, and dermal contact [170, 171]. The utilization ad application of pesticide in Egypt needs more regulations and desires greater attention. Therefore, treatment of pesticides-polluted water is of great importance.

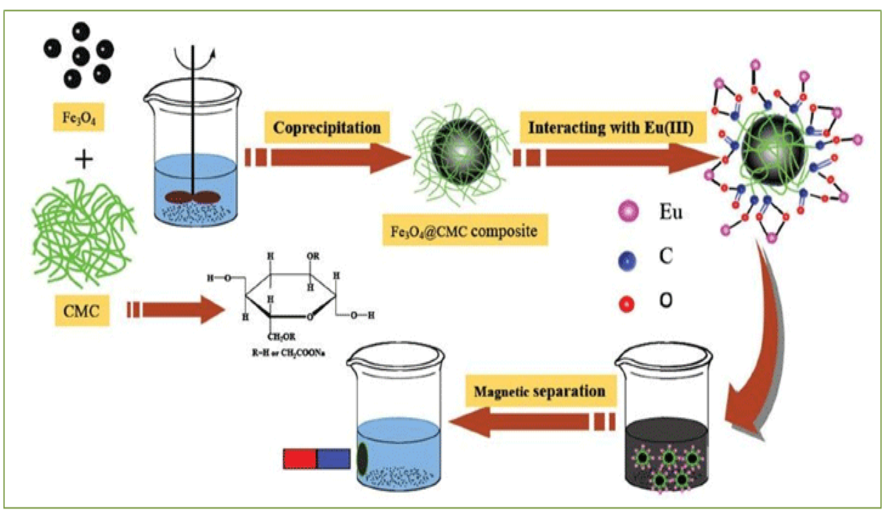

Figure 21. Illustration for the synthesis and removal mechanisms of Fe3O4@CMC composite toward Eu(III) [165]

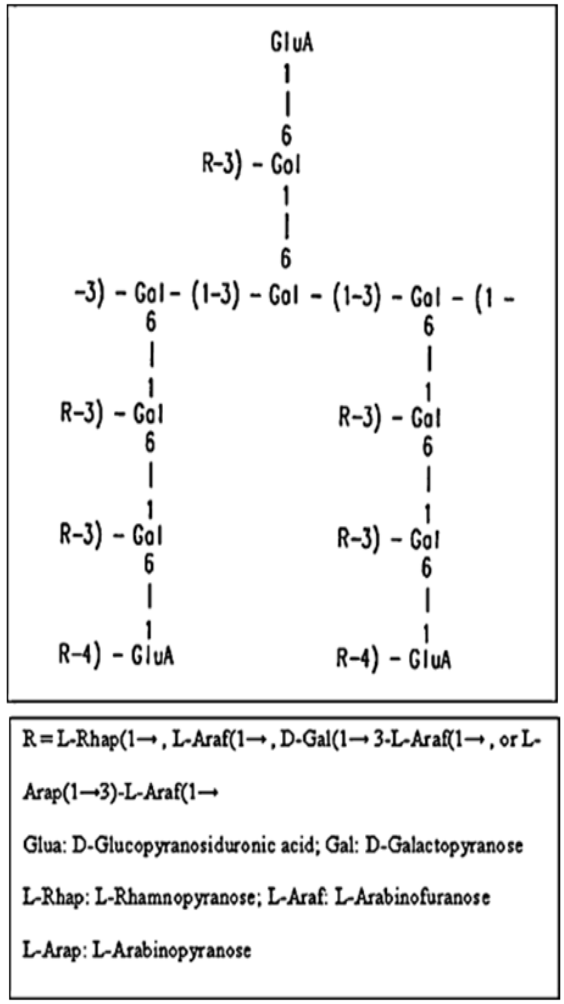

Figure 22. Chemical structure of Arabic gum

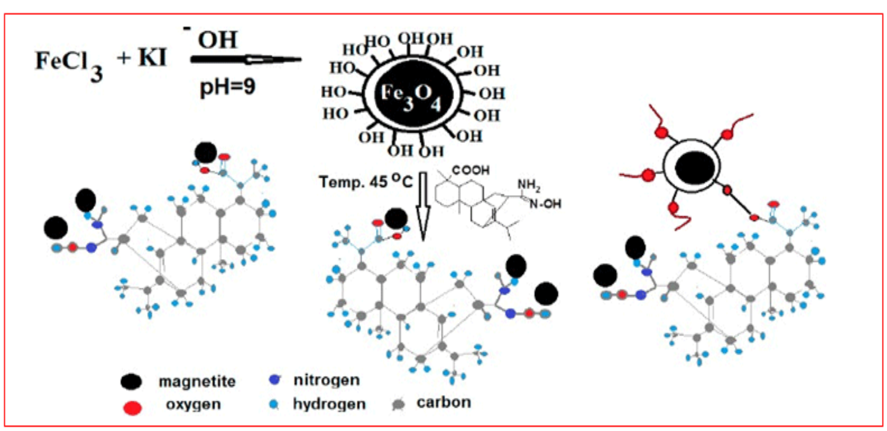

Figure 23. Synthesis of magnetite coated rosin amidoxime.

Conclusions

The problem of water deficiency was reviewed. Types of water pollutants have been discussed. Furthermore, conventional and modern methodologies applied for wastewater treatment were investigated in details with comparison between the advantages and disadvantages of each method. The review concentrated on adsorption as the best method applied in wastewater treatment dealing with the definition of adsorption and listing types of different adsorbents. The superiority of hydrogels over other types of sorbents was explained. Hydrogel nanoparticles and polymer nanocomposites were deeply investigated shedding light on natural polymers used as polymeric shields for the nanocomposites such as β cyclodextrine, starch, chitosan, guar gum, carboxymethyl cellulose, and Arabic gum. The utilization of some agricultural wastes such as orange peels and rice husk in removal of some heavy metals was studied.

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