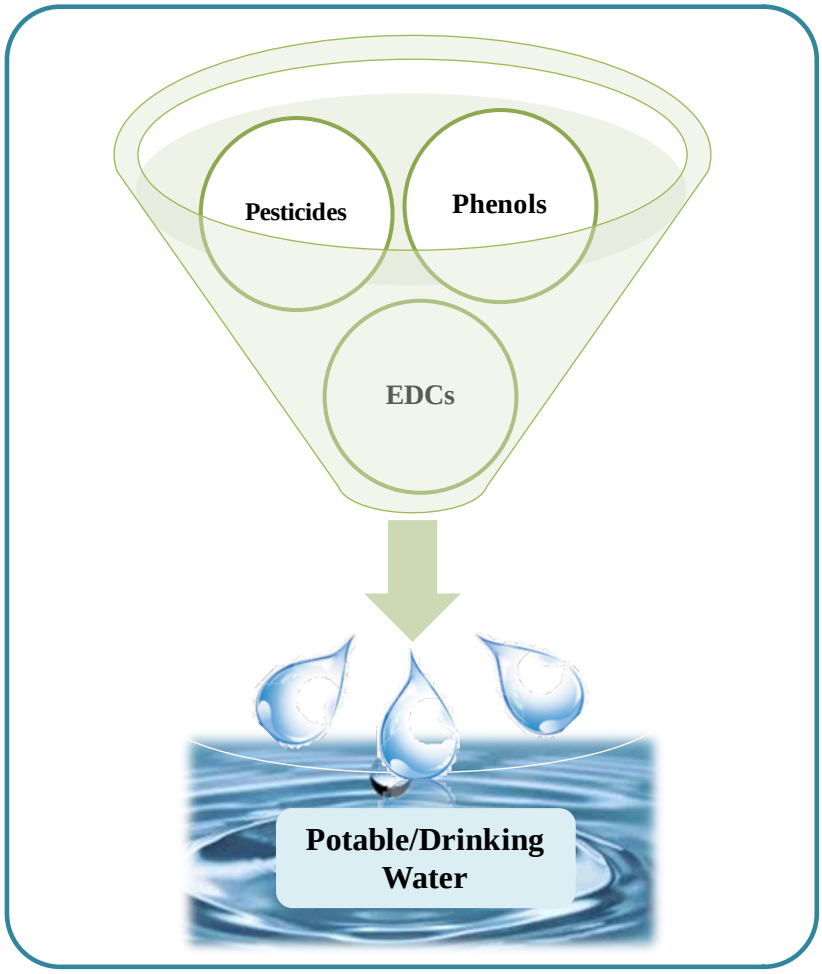
Chapter 1

Introduction



1.1 Introduction

Contamination of water resources by hazardous chemicals and endocrine disrupting compounds (EDCs) is widespread and is a topic of special environmental relevance, due to the toxicity of these substances (Cai et al., 2003). For example, phenols and substituted phenols are toxic organic pollutants commonly present in industrial effluents especially in industrial wastewater from oil refineries, coal gasification, petrochemicals, polymeric resins, coal tar distillation, pharmaceuticals, etc (Rajkumar and Palanivelu, 2003). Chlorinated phenols are an important class of aromatic pollutants that exist in industial wastewaters because of their wide use in the production of pesticides and biocides. Major sources of environmental contamination by chlorophenols are the widespread use of pentachlorophenol as a wood preservative, degradation products of chlorinated herbicides, chlorination of lignin or the use of slimicides in paper or pulp mill plants, chlorination of municipal and industrial wastewater and drinking water, wastewaters from petrochemical units, coal gasification sites, oil refineries and pharmaceutical industries (Czaplicka, 2004; Exon, 1984). Chlorophenol (CP) is primarily listed as one of the priority pollutants (Czaplicka, 2004) because of its high toxicity, strong odor and persistence in the environment as well as suspected carcinogenity and mutagenity to living organisms (US EPA, 1987). If the chlorophenol containing effluents are not treated properly in industial wastewater treatment plants, their strong toxicity, persistence in the environment and suspected carcinogenecity may pose serious environmental problems (Quan et al., 2005).

Nitro-aromatic compounds, especially nitrophenols, are widely used in chemical industry, e.g. in the production of dyes, pesticides, pharmaceuticals, explosives, petrochemicals, petroleum refineries, coke oven, steel foundry, insecticides, herbicides and leather treatment units (Shen et al., 2009).

4-Nitrophenol (4-NP) is another priority pollutant widely used for the manufacture of drugs, dyes, fungicides, insecticides and to darken leather. Effluents from the textile industry, waste waters from various industries such as iron and steel manufacturing, foundries, pharmaceutical manufacturing, processing and electrical/electronic components production may also release both 2-nitrophenol and 4-NP into the surface

waters. The compound, trinitrophenol (TNP) or picric acid, is particularly important, because it is an intermediate in the manufacture of explosives that are more powerful than the well known explosive, 2,4,6-trinitrotoluene (TNT). Nitro aromatic compounds, including NPs, are used in the production of dyes (Aktaş et al., 2007). Due to their toxic and carcinogenic nature, they pose a threat to animals and plants in the biosphere necessitating compulsory removal of these compounds from the effluents before safe disposal into the surface waters (Kumar et al., 2007) and they are listed as priority pollutants by the United States Environmental Protection Agency (EPA) (Rawajfih and Nsour, 2006).

EDCs are substances that mimic natural hormones in the endocrine system and thus cause adverse effects on humans and wildlife (Wong et al., 2016). Bisphenol A [2,2-bis(4-hydroxyphenyl)propane] (BPA), an EDC with a weak estrogen-like effect has caused great concern because of its potential risk to human health (Ou et al., 2016; Pojana et al., 2007). BPA and 4-NP are widely used as raw materials and intermediates for epoxy and polycarbonate resins (Ding et al., 2015) which are widely used in different products. BPA has also been used as an inert ingredient in pesticides, antioxidants, flame retardants, rubber chemicals and polyvinyl chloride stabilizers. BPA can enter a body of water during manufacturing, leach from plastic products, and be disposed in landfills after use (Šala et al., 2010).

Monocrotophos (dimethyl (*E*)-1-methyl-2-(methylcarbamoyl) vinyl phosphate) (MnCP) is an organo phosphorus pesticide. It is very toxic to birds and is used as a bird poison. It is used to control a variety of pests on cotton, sugar cane, peanuts, ornamentals, and tobacco (Kidd and James, 1991). It is also very harmful to mammals. Cases of human poisoning are characterized by muscular weakness, blurred vision, perspiration, vomiting, pain, and small pupils. There is a risk of death due to respiratory failure (Gupta, 2004). The EPA classifies MnCP to be of class I toxicity (Willoughby O.H., 1994).

The effective removal of these toxic and priority pollutants from water is an issue of great importance and interest due to their toxicity. Further, because of the accumulation and vast scale distribution of NPs, CPs, BPA and MnCP in the ecological environment, their

separation and determination have become topics of intense research in environmental analysis.

These issues emphasize the importance of developing reliable, highly sensitive, user-friendly and cost effective techniques for the removal as well as constant monitoring and detection of pollutants in different media which still remains a challenge.

1.2 Remediation techniques

Conventional methods for the removal of organic pollutants in effluents may be divided into three main categories: physical, chemical, and biological (Hao et al., 2000). Complete removal of EDCs and other aromatics like CP was not achieved through biological processes as they are recalcitrant to biodegradation. To achieve complete satisfactory elimination of organic pollutants, further advanced treatment is needed. Therefore, techniques like photo degradation, dechlorination, electrochemical oxidation, ultra filtration, wet oxidation, solvent extraction, membrane separation, and adsorption have been proposed as possible treatment methods (Catrinescu et al., 2011; El-Sheikh et al., 2011; Elghniji et al., 2012; Murcia et al., 2009; Su et al., 2011; Sze and McKay, 2010; Wang et al., 2011; Duan et al., 2012). However, these methods suffer drawbacks such as requiring large energy input, slow process or use of chemicals (İpek et al., 2014). Ozonation processes can produce by-products that are undesirable for water treatment (Hübner et al., 2014). ***Adsorption*** and ***Chemical advanced oxidation*** techniques are generally used for the removal of BPA, NPs, CPs and MnCP. Amongst them, adsorption has been found to be superior in terms of ease of operation as well as lower operating cost, and the lack of the possibility of producing secondary harmful products (Zheng et al., 2013). Further adsorption processes are cost-effective, can be used for a wide range of pollutant concentrations and are amenable for recycling of pollutants (Tobajas et al., 2012).

1.2.1 Adsorption

In recent years, research has been focused on the study of techniques based on adsorption approaches using low–cost and environmentally friendly adsorbents for the elimination of organo pollutants In this context, great research efforts have been directed towards

adsorption processes and adsorbent materials generally possessing large accessible internal and/or external surface for the pollutants to be adsorbed (Khalid et al., 2004).

I) Biosorbents and other low cost sorbents

The adsorption of phenols by low cost adsorbents was reviewed by Ahmaruzzaman (Ahmaruzzaman, 2008).

The sorption behaviour of BPA from aqueous solutions onto a biosorbent such as peat, rice husk, bagasse, and sawdust was evaluated (Zhou et al., 2012). Nadavala et al. used chitosan-calcium alginate blended beads for biosorption of phenol and o-CP (Nadavala et al., 2009). The adsorption behavior of two organophosphorus pesticides MnCP and chloropyrifos onto Acid-Treated Palm Shell Powder prepared from palm shells was investigated by Kushwaha et al. (Kushwaha et al., 2011). The biosorption of phenols was reviewed by Mathialagan and Viraraghavan (Mathialagan and Viraraghavan, 2008). Modern technologies for the removal of phenol from fluid streams were reviewed by Busca et al. (Busca et al., 2008). Denizli et al. reported removal of CPs from aquatic systems using dried and dead fungus Pleurotus sajor caju (Denizli et al., 2005). Bayramoglu et al., reported the use of Funalia trogii pellets for biosorption of phenol and 2-CP (Bayramoglu et al., 2009). Lang et al. used dead biomass of Rhizopus arrhizus encapsulated in chitosan beads for the biosorption of nonylphenol (Lang et al., 2009). Vazquez et al. reported adsorption of phenol on formaldehyde-pretreated Pinus pinaster bark (Vázquez et al., 2007). Marine sea weeds and algae are also of great interest for biosorption (Aravindhan et al., 2009; Navarro et al., 2008).

Adsorption of BPA on sediments has been studied by Zeng et al. (Zeng et al., 2006). Sewage waste (activated sludge and inactivated sludge) was converted into adsorbent by Clara et al. and was used for the adsorption of BPA and estradiols (Clara et al., 2004). Removal of BPA from aqueous solution by hydrophobic sorption of hemimicelles was investigated by Gong and coworkers (Gong et al., 2009). Sludge biomass (Seyhi et al., 2011), chitosan (Gong et al., 2010) and Fe (III)/ Cr (III) hydroxide (Namasivayam and Sumithra, 2007) have been used for the removal of phenols.

II). Zeolites

Zeolites are an important class of hydrated aluminosilicates. They possess cage-like structures with internal and external surface areas of up to several hundred square meters per gram. An important property of these materials is the capacity to be regenerated while retaining their initial properties.

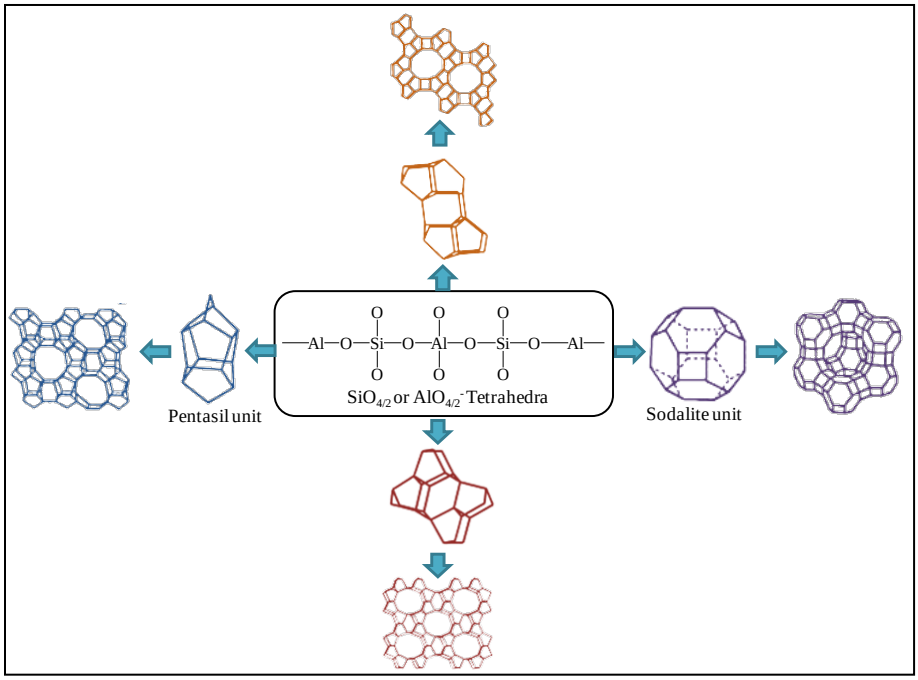


Figure 1.1 Structure of Zeolie

Synthetic and natural zeolites have been widely used as adsorbents for removal of phenolic compounds from wastewater (Gómez-Hortigüela et al., 2014; Huong et al., 2016). A hydrothermally synthesized zeolite was investigated for its adsorption potential towards methylene blue and BPA (Tsai et al., 2009). Zeolite synthesized from coal fly ash was modified with hexadecyltrimethylammonium (HDTMA) and was examined for the adsorption of BPA from water by Dong et al., (Dong et al., 2010). Genc et al., used surfactant (HDTMA)-modified natural zeolite to remove BPA from aqueous solutions (Genc et al., 2017).

III). Montmorillonite

Among naturally abundant materials, clay minerals are potential adsorbents due to their low cost, environmental stability, high adsorption/absorption, and ion exchange properties. In particular, montmorillonite (MMT) is widely used because of its high

cation exchange capacity (CEC), swelling property, and high surface area (Park et al., 2011). However, one of the drawbacks of clay minerals is their hydrophilic properties which make them ineffective for the removal of organic pollutants. This drawback is often overcome by modifying the clay with organic cations. For instance, organo-montmorillonite has been used for removal of BPA (Park et al., 2014).

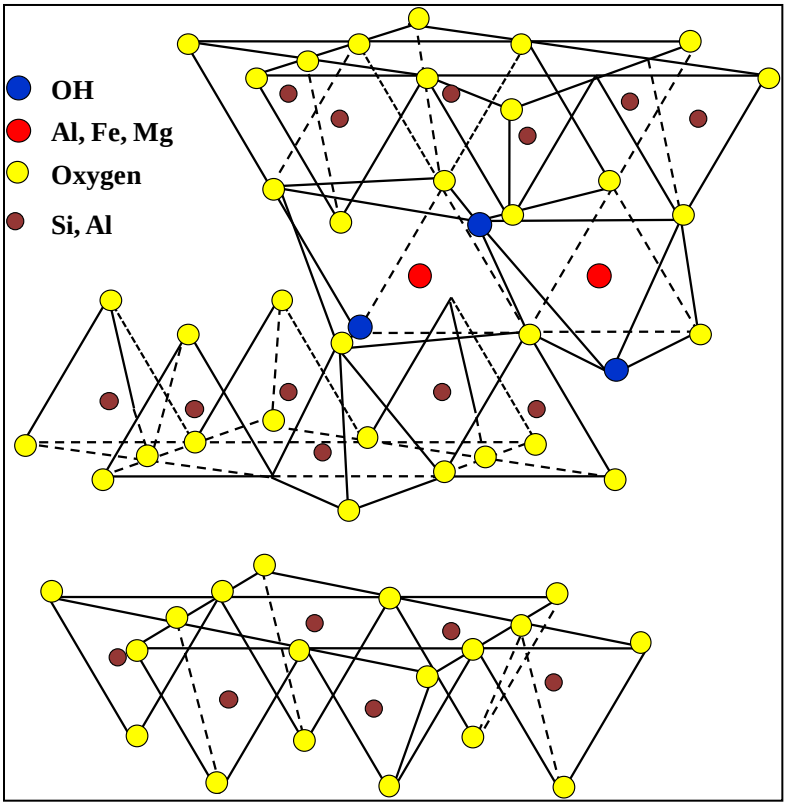


Figure 1.2 Structure of Montmorillonite

The surface properties of Ca-MMT were changed from hydrophilic to hydrophobic through intercalation with DDTMA (Dodecyltrimethyl ammonium bromide) and HDTMA (Hexadecyltrimethyl ammonium bromide) were used for removal of BPA by Zheng et al. (Zheng et al., 2013). Remediation of BPA from aqueous solutions by adsorption using organoclays synthesized from MMT with different organic surfactant molecules was demonstrated by Park et al. (Park et al., 2014).

To utilize the π–π interaction as well as the hydrophobic affinity of modifiers with long alkyl chain in the interlayer of montmorillonites for effective removal of BPA from aqueous solution, organo-MMTs modified with gemini pyridinium surfactants containing

both the aromatic ring and long alkyl chain, 1,1′-didodecyl-4,4′-trimethylene bispyridinium bromide (BPy-12-3-12) and 1,1′-dihexadecyl-4,4′-trimethylene bispyridinium bromide (BPy-16-3-16) were designed by Yang et al. (Yang et al., 2016). Polyethersulfone-organophilic MMT hybrid particles were used for adsorption of BPA (Cao et al., 2009). Removal of 4-NP from aqueous solution by Organo-MMTs modified with ionic liquids was investigated by Baysal (Baysal, 2016). MMTs modified by two gemini surfactants (1,3-bis(dodecyldimethylammonio)-propane dibromide and 1,3-bis(dodecyldimethylammonio)-2-hydroxypropane dichloride via ion exchange were used to remove 4-NP from aqueous solutions by Xue et al., (Xue et al., 2013). Palygorskite was modified with HDTMA by Zhao et al. (Zhao et al., 2014) to make it amenable for BPA removal.

IV). Biochars

Biochar is obtained as a byproduct of the pyrolytic processing of biomass when biofuel is produced during controlled thermal processes and gasification. Biochar shows potential as a promising adsorbent for the removal of micropollutants due to its increased surface density of functional groups and highly condensed structure. These properties vary depending on the type of feedstock, pyrolysis conditions (residence time and temperature), and activation. In particular, while higher proportions of aliphatic carbons and functional groups are typical of biochars pyrolyzed at low temperatures, biochar pyrolyzed at higher temperatures contains mainly polyaromatic carbons and has a higher microporosity, which enhances organic pollutant adsorption (Chen et al., 2008; Chun et al., 2004). The adsorption of sunscreen compounds (benzophene (BZP) and benzotriazole (BZT)) and EDCs (BPA and 17β-estradiol (E2)) was investigated using commercially available PAC and activated biochar from pine chips (Kim et al., 2016).

V). Coal

Removal of EDCs such as amitrol, nonylphenol, and BPA was investigated using coal (Choi et al., 2005) and fibric peat (Zhou et al., 2011).

VI). Activated Carbon

Activated carbon has a special place among adsorbents due to its unique non polar or slightly polar surface property (e.g., clay) (Yang, 1987). In this regard, activated carbon

is now the most widely used adsorbent for removal of organic molecules. Currently, activated carbons are manufactured from a variety of starting materials.

The adsorption of BPA on different activated carbons was observed to be strongly dependent on the chemical nature of the carbon surface and the solution pH by Toledo et al. (Bautista-Toledo et al., 2005). The authors also observed that the adsorbent-adsorbate interactions governing the adsorption mechanism are enhanced when the net charge density of the carbon is zero and the BPA is in molecular form.

The adsorption kinetics of nine contaminants (Ibuprofen, Carbamazepine, Ofloxacin, BPA, diclofenac, mecoprop, pentachlorophenol, benzotriazol and caffeine) on a microporous and a microporous/mesoporous activated carbon cloth were studied by Masson et al. in single, two-component and complete mixture at pH 7.5 (Masson et al., 2016). Two commercial carbons (W20 and F20) had been selectively modified with nitric acid and thermal treatment under a flow of N_2 by Liu et al. to adsorb BPA from aqueous solution with adsorption capacity reaching up to 432 mg g^{-1} (Liu et al., 2009).

Aktas and Cecen studied the effect of four different types of activated carbons; thermally activated and chemically activated powdered carbons (PAC) and their granular forms (GAC) with similar physical characteristics on the extent of adsorbability, desorbability, and bioregenerability in the removal of 2-CP. Thermally activated carbons adsorbed 2-CP relatively better than chemically activated ones though adsorption was more reversible in the case of chemically activated ones (Aktaş et al., 2007).

However, the cost of activated carbons is exorbitant due to the use of non-renewable precursors and activation techniques adopted. Nowadays, research has been focused on the use of industrial and agricultural wastes as precursors for preparation of low cost carbon with added advantages of low treatment time and low energy consumption to generate high volume product.

Agrowaste derived carbon

Among sorption materials, waste carbonaceous material-based AC has been studied extensively as it significantly reduces production costs. Many industrial and agricultural wastes have been used as renewable precursors for activated carbons such as, pistachio

shell, coconut shell, and fertilizer waste for the removal of chlorophenols and other phenols (Radhika and Palanivelu, 2006; Tseng et al., 2010). Ahmed et al. used Albizia lebbeck seed pods as precursors for production of a microporous activated carbon by microwave assisted K_2CO_3 activation and investigated its sorption potential for 4-CP (Ahmed and Theydan, 2013).

Bautista-Toledo et al. studied the adsorption of BPA from water on two commercial activated carbons and almond shells derived carbon and observed that the adsorption process depended on the chemical nature of the carbon surface and the pH of the solution. The presence of mineral matter in carbons reduced their adsorption capacity because of the hydrophilic nature of the mineral matter (Bautista-Toledo et al., 2005). They further investigated the simultaneous adsorption of BPA and chromium ions from aqueous solution on activated carbons (both commercial and prepared from olive-mill waste) (Bautista-Toledo et al., 2014). The adsorption behavior of BPA, from aqueous solution onto four minerals and two activated carbons derived from coconut and coal were examined by Tsai et al. They observed that the adsorption capacities of activated carbons were significantly larger than those of mineral adsorbents, implying that the former is effective for removal of hydrophobic BPA because of its high surface area and low surface polarity (Tsai et al., 2006). Nakanishi et al. investigated the adsorption of BPA on carbon derived from wood chips (Nakanishi et al., 2002). The potential of the activated carbon prepared from the empty fruit bunch of oil palm wastes to remove BPA from aqueous media was investigated by Wirasnita et al. (Wirasnita et al., 2014). Triethoxyphenylsilane functionalized magnetic palm-based powdered activated carbon was prepared and investigated for the removal of BPA, carbamazepine, ibuprofen and clofibric acid (Wong et al., 2016).

Asada et al., (Asada et al., 2004) used porous carbon produced from the carbonization of bamboo as an effective adsorbent for removal of BPA from aqueous solution. They observed that the degree of adsorption of BPA increased as the carbonization temperature increased, due to the corresponding increase in BET surface area. They also observed that carbon adsorbent with a low surface polarity would be more effective for the adsorption of hydrophobic BPA.

Sorbo Norit (obtained by water steam activation of peat) and Ceca AC40 (produced from maritime pinewood chemically activated with phosphoric acid),were used as adsorbents for single, competitive and dynamic adsorption of phthalic acid , BPA, diphenolic acid, 2,4-dichlorophenoxy-acetic acid (2,4-D), and 4-chloro-2-methylphenoxyacetic acid by Abdel Daiem et al. (Abdel Daiem et al., 2015). Olorundare et al. used maize tussel derived activated carbon cartridge packed solid phase extraction (SPE) system for both assay and remediation of BPA, o-Nitrophenol (2-NP) and 4-CP effectively (Olorundare et al., 2015).

The adsorption of 4-CP from aqueous solution on activated carbon derived from rattan sawdust has been investigated by Hameed et al. (Hameed et al., 2008). Activated carbons with high adsorption capacities for chlorophenols were fabricated from fir wood (Wu et al., 2005). Senthilkumar et al. studied the adsorption of monocrotophos from aqueous solution using “waste” jute fiber carbon (Senthilkumaar et al., 2010). Activated carbon can be applied in powdered (PAC) or granular (GAC) form.

Powder Activated Carbon

The ASTM classifies particles passing through an 80-mesh sieve (0.177 mm) and smaller as PAC. A pilot study was performed by Mailler et al. using fluidized PAC (CarboPlus) and 54 pharmaceuticals, 30 hormones and 59 other emerging pollutants including BPA were monitored in influents and effluents of the pilot plant (Mailler et al., 2015). Gong et al. oxidised commercial PAC by two methods, wet oxidation with ammonium persulfate and thermal treatment after acidification with hydrochloric acid and further functionalized them with thermoresponsive poly (N-isopropylacrylamide) (PNIPAM) in aqueous solution at ambient temperature. The oxidized PAC modified with thermal treatment after acidification as well as its derivative grafted with PNIPAM showed larger surface area and better adsorption of BPA. In addition, the grafted PAC products show self-flocculation behaviors with rapid response to temperature because of the thermal phase transition and entanglement behaviors of PNIPAM (Gong et al., 2016).

PACs (Norit DARCO A-51, USA), impregnated with iron oxide nanoparticles were investigated for the simultaneous removal of BPA and NOM (Park et al., 2015).

Granular Activated Carbon

GAC has a relatively larger particle size compared to powdered activated carbon and consequently, presents a smaller external surface. GAC is designated by sizes such as 8×20, 20×40, or 8×30 for liquid phase applications. A 20×40 carbon is made of particles that will pass through a U.S. Standard Mesh Size No. 20 sieve (0.84 mm) (generally specified as 85% passing) but be retained on a U.S. Standard Mesh Size No. 40 sieve (0.42 mm) (generally specified as 95% retained) (Saksule and Kude, 2012).

Removal performances of EDCs such as amitrol, nonylphenol, and BPA were evaluated by several researchers using GAC (Choi et al., 2005; Pan et al., 2008). GAC Filtracarb CC60 was used for the removal of BPA (Katsigiannis et al., 2015). Sudhakar et al., compared adsorptive removal efficiencies of low-cost adsorbent rice husk ash (RHA) and commercial GAC for the removal of BPA, wherein GAC was found to be better than rice husk ash (Sudhakar et al., 2016). GAC has been observed to be one of the efficient adsorbents for removal of CPs from wastewaters because of its uniform pore size distribution and high microporous content (Oh et al., 2011), Choi et al. examined the removal performances of three endocrine disrupting chemicals using GACs with three different base materials. They observed that the pore volume of carbons was more critically affecting the adsorption capacity than the specific surface area and that surface charge also played a role probably due to the electrical interaction between the nearly non-polar carbon surface and highly hydrophobic molecule BPA(Choi et al., 2005). Liu et al. investigated the adsorption of BPA on GACs with different surface chemical properties and observed a BPA adsorption capacity of up to 432 $mg.g^{-1}$, which was lesser with higher temperature and solution pH values (Liu et al., 2009).

However, PAC is better than that of GAC because of its larger surface areas (Tsai et al., 2006). But the small particle size makes the retrieval of PAC from water difficult, and the residual PAC in the water will result in the secondary pollution (Gong et al., 2016).

Biological Activated Carbon

A biofilm on activated carbon (biological activated carbon) is formed for effective removal of pollutants. Serratia rubidiae, Pseudomonas aeruginosa and Escherichia coli

K12 have been studied for their ability of BPA removal from aqueous systems and biofilm formation on GAC (Mita et al., 2015)

Sewge Sludge Derived Carbon

Activated carbons of different characteristics have been prepared from dried sewage sludge using CO_2, air and KOH as activating agents. The adsorption capacity of the resulting materials has been checked using 4-CP as a target compound by Monsalvo et al. (Monsalvo et al., 2011).

VII). Ordered mesoporous carbon

Ordered mesoporous carbon (OMC) with the characteristics of high chemical stability, large specific surface area, production feasibility was regarded as a promising adsorbent for BPA removal (Sui et al., 2011). Mesoporous carbons were synthesized by Libbrecht et al. via both soft and hard template methods and were compared with commercial PAC for the adsorption ability of BPA from an aqueous solution. Soft template method had higher adsorption capacity. The maximum observed adsorption capacity (qmax) of CMK-3 was 474mg.g^{-1}which was higher compared to 290mg.g^{-1} for PAC (Libbrecht et al., 2015). Sui et al. also investigated the adsorptive removal of BPA using CMK-3 with high surface area of 920m^2.g^{-1} (Sui et al., 2011).

Liang and Dai performed soft templated synthesis of ordered mesoporous materials by self-assembly of tri-block copolymer surfactants and carbon precursor (Liang and Dai, 2006) while Kruk et al. synthesized hard templated ordered mesoporous carbon using mesoporous carbon as template (Kruk et al., 2000).

Iron nanoparticles doped magnetic ordered mesoporous carbon (Fe/OMC) were prepared by co-impregnation and carbothermal reduction techniques and used for efficient adsorption and degradation of BPA (Tang et al., 2016). The degradation was based on Fenton reaction where zero-valent iron was oxidized into ferrous ions by oxygen to produce hydrogen peroxide, then the hydrogen peroxide and bivalent iron oxidized BPA. Tripathi et al. prepared hierarchically ordered micro–mesoporous carbon with enlarged uniform micropores, specifically tailored for the high adsorption (1106 mg.g^{-1}) of BPA. Sizes of both the primary micropore (1.3 nm) and the primary mesopore (9.0 nm) could be tuned by controlling the condensation behavior of phloroglucinol-terephthalaldehyde

resin in a tri-constituent system based on evaporation induced self-assembly (Tripathi B.P. et al., 2014).

VIII). Carbon nanomaterials

Carbon nanotube

Since carbon nanotubes (CNTs) were discovered by Iijima in 1991 (Iijima, 1991), they have aroused wide attention due to their unique and superior physicochemical properties. CNTs exhibited a very strong affinity for both organic contaminants and heavy metal ions seeming to be superior and promising sorbents applied in wastewater treatment (Bohdziewicz and Kaminska, 2013; Jung et al., 2015; Ren et al., 2011; Yu et al., 2014), due to their unique extremely large surface area and highly hydrophobic surfaces (Strachowski and Bystrzejewski, 2015). Fluorinated CNTs have been used by Li et al., for removal of sulfamethoxazole, ofloxacin, norfloxacin, BPA and phenanthrene (Li H. et al., 2016). Advantage was taken of the electronegative nature of F in C-F bond enabling strong π-π interactions during adsorption of organic compounds. In addition, the C-F bond can hardly form hydrogen bond with water molecules further enhancing the adsorption of hydrophobic compounds.

The feasibility of BPA adsorption from aqueous solutions by SWCNTs and MWCNTs was assessed by several workers (Dehghani et al., 2015; Pan et al., 2008). Commercial SWCNTs were used by Zaib et al., for removal of BPA and estradiol (Zaib et al., 2012). Polyacrylonitrile membranes modified with SWCNTs have ben used for removal of nonylphenol and BPA (Kamińska et al., 2016).

MWCNTs also exhibited good affinity towards several organic pollutants including BPA (Dai et al., 2016; Wang W. et al., 2014). MWCNTs as adsorbents have attracted increasing attention due to their higher specific surface area, high reactivity, large micropore volume and unique hollow tube structure. Because of their distinctive properties of sp^2 hybridized carbon bonds, MWCNTs have exhibited great adsorptive affinity to a series of phenolic pollutants from aqueous solutions (Yu et al., 2014) and found to be efficient adsorbents with a capacity that exceeds that of activated carbon.

Recently, most adsorption studies have focused on MWCNTs, due to their being more economic than SWCNTs. However, SWCNTs generally have higher adsorption capacity

than MWCNTs, due to their larger specific surface area and smaller diameter(Apul and Karanfil, 2015).

Exfoliated graphite nanoplatelets

Exfoliated graphite nanoplatelets were used as adsorbents for BPA (Radu et al., 2015).

Graphene:

Graphene, an environmental friendly material, which generally has high thermal and electrical conductivities, thermal stability and mechanical flexibility (Balandin, 2011; Huang et al., 2012; Zhu et al., 2010) was first reported in 2004 by Andre Geim and his colleague Kostya Novoselov (Novoselov et al., 2004). It is a single atomic layer of sp^2-hybridized carbon arranged in a honeycomb structure, is the 2D allotrope of carbon. It has drawn enormous scientific attention since its discovery because of its large surface area (Lee et al., 2008). Compared to other carbonaceous adsorbents, the advantage of graphene is its selective adsorption ability towards aromatic compounds with benzene rings through strong π-π interactions.

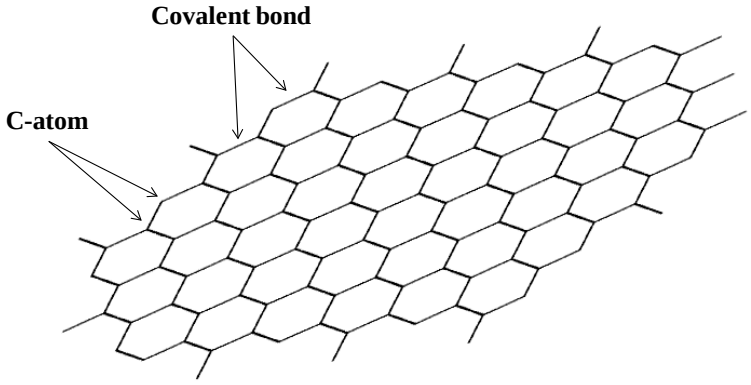


Figure 1.3: Structure of Graphene

Graphene and chemically modified graphene forms such as graphene oxide (GO) and reduced GO (rGO) are thus expected to be promising adsorbents for the removal of aromatic compounds in water treatment (Lee et al., 2008). The chemical oxidation of graphite with the Hummers method ($KMnO_4$, $NaNO_3$, and H_2SO_4) is the commonly used method for preparing GO (Marcano et al., 2010). GO can be reduced to rGO by using suitable chemicals or biological reducing agents, such as hydrazine hydrate and sodium borohydrate, glucose, ascorbic acid, and galactose (Gao et al., 2010).

For instance, graphene has been used as an adsorbent for the removal of BPA from aqueous solutions (Kwon and Lee, 2015). Adsorption of three selected pharmaceuticals and personal care products ketoprofen, carbamazepine and BPA by two rGOs and one commercial graphene was investigated by Liu et al. SWCNTs, MWCNTs and powdered graphite were also investigated as adsorbents for comparison (Liu et al., 2014).

Carbon nanofibers

Carbon nanofibers have advantage of chemical and thermal stability, large surface area and narrow pore distribution. CNFs were prepared by electrospun polyacrylonitrile polymer solutions followed by thermal treatment and have been used for adsorption of ciprofloxacin, BPA and 2-CP (Liu et al., 2014).

IX). Polymeric adsorbents

Polymeric adsorbents have gained importance (İpek et al., 2014; Wagner and Schulz, 2001) recently. Currently, advances in polymer science have provided opportunities to select well-structured polymeric adsorbent with high surface area, pore size, and suitable surface-chemistry, such as hypercrosslinked polymeric adsorbents and aminated polymeric adsorbents (Ahn et al., 2006; Penner and Nesterenko, 2000; Xiao et al., 2012). Moreover, one of the main advantages of these polymeric adsorbents is that it can be regenerated more easily than other materials by using organic solvents such as ethanol, methanol, or acetone (Valderrama et al., 2007). Fan et al. evaluated porous hypercrosslinked aminated resins as adsorbents for removal of phenol, BPA and nonylphenol ethoxylates from aqueous solutions (Fan et al., 2011). Polysulfone containing membranes exhibited very high adsorption ability for BPA (Su-Hua et al., 2010).

Cyclodextrin polymers

β-Cyclodextrin (β-CD), which possesses a hydrophilic exterior and a hydrophobic cavity, is a water-soluble macrocyclic oligomer of d-(+)-glucopyranosyl units linked by α-1,4-glycosidic bonds, is readily available from the enzymatic degradation of starch, and possesses excellent biocompatibility (Abdel-Naby et al., 2011).

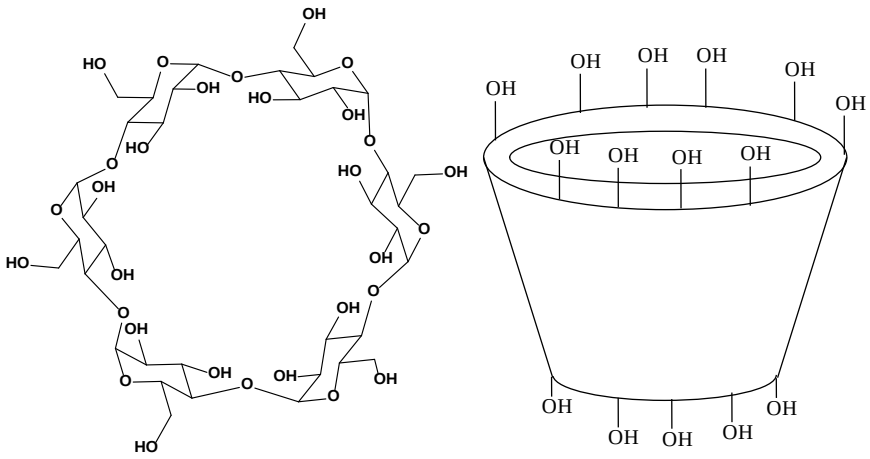


Figure 1.4 Structure of cyclodextrin

β-CD is known to encapsulate pollutants to form well-defined host–guest complexes. Further, some researchers reported the synthesis of the polymers having the CD moiety and demonstrated their potential to remove toxic compounds from water by means of adsorption (Morin-Crini and Crini, 2013; Szejtli, 1988). However, most of the cross-linked β-cyclodextrin polymers were reported with low surface areas and poor removal performance compared to conventional activated carbons (Crini and Morcellet, 2002; Lo Meo et al., 2014). Alsbaiee et al. crosslinked β-CD with rigid aromatic groups using tetrafluoroterephthalonitrile, providing a high-surface-area, mesoporous polymer of β-CD and has been used for removal of 4-CP, BPA and triclosan (Alsbaiee et al., 2016). An insoluble crosslinked chitosan bearing CD moieties was prepared by a one-step procedure with N-succinylated chitosan and mono-6-amino-mono-6-deoxy- β-CD in the presence of the water-soluble carbodiimide for adsorption of BPA (Aoki et al., 2003).

Bhattarai et al. reported removal of several ECs from water including 17β-estradiol, perfluorooctanoic acid, and BPA by β-CD coated on silica, synthesized using two different crosslinking agents (hexamethylene diiso-cyanate and epichlorohydrin) and co-polymers (glycidoxypropyl trimethoxysilane and aminopropyl triethoxysilane) (Bhattarai et al., 2014). The suitability of a CD-based bead polymer, for modelling the removal of micropollutants from drinking water and purified waste water using simulated inflow test solutions containing target analytes ibuprofen, naproxen, ketoprofen, BPA, diclofenac, β-estradiol, ethinylestradiol, estriol, cholesterol at 2–6 $\mu g.L^{-1}$ level was investigated by Nagy et al. (Nagy et al., 2014).

Molecularly imprinted polymers

Molecularly imprinted polymers (MIPs) are synthetic polymers which are formed in the presence of a target molecule that finally is removed by the use of proper solvents; therefore, the obtained specific cavities are complement to the target molecule in shape, size and functional groups (Mehdinia et al., 2013). In recent years, the combination of MIP and other sample preparation techniques like solid phase extraction, solid-phase microextraction and matrix solid-phase dispersion has opened a new window for selective extraction and recognition of target molecules from the complex matrices (Turiel and Martín-Esteban, 2010). An analytical procedure for the selective extraction and detection of 4-NP was investigated by using MIP on the surface of magnetic nanoparticles (Mnps). The Mnps were modified by tetraethyl orthosilicate and 3-methacryloxypropyl trimethoxysilane before imprinting. The magnetic MIP was polymerized at the surface of modified Mnps by the use of methacrylic acid as functional monomer, 4-NP as template and ethylene glycol dimethacrylate as cross-linker. The assay exhibited a linear range of 25–1000 μg L^{-1} for 4-NP (Mehdinia et al., 2013).

An MIP with 4-chlorophenyl (4-vinyl) phenyl carbonate as template, ethylene glycol dimethacrylate as cross-linker, 2,2-azobisisobutyronitrile, and chloroform as initiator and porogen, respectively was investigated for removal of phenol, 4-CP, 2,4-DCP, 2,4,6-TCP (Qi et al., 2010). Molecularly imprinted polystyrene divinylbenzene as well as kaolinite-polystyrene divinylbenzene based MIP adsorbents were used for adsorption of BPA (Guo et al., 2011; Laatikainen et al., 2014).

Novel magnetic MIPs based on multiwalled carbon nanotubes (MWCNTs@MMIPs) with specific selectivity towards BPA were synthesized using BPA as the template molecule, methacrylic acid, and β-cyclodextrin as binary functional monomers and ethylene glycol dimethacrylate as the cross-linker (Zhang Y. et al., 2014).

X). Composites

Nowadays, considerable efforts have been made to produce metal-carbon/polymer nanocomposite materials, not only because the carbon compound improves the mechanical properties of the composites, but also because the produced composites possess the properties of individual components with a synergistic effect

(Mohammadkhani et al., 2015; Zhang Z. et al., 2014) Sahithya et al. used biopolymer modified MMT–CuO composites viz. MMT–CuO–Chitosan, MMT–CuO–Gum ghatti, and MMT–CuO poly lactic acid as adsorbents for removal of MCP attaining an adsorption capacity of 212.23 mg g^{-1} (Sahithya et al., 2016). A magnetically separable $CoFe_2O_4$/PAC composite adsorbent has been prepared by Li et al. (Li Z. et al., 2014) for the removal of BPA. Magnetic CNTs/Fe_3O_4 nanocomposites in which CNTs were hydrothermally grafted to magnetic Fe_3O_4 particles have been used for removal of BPA (Li S. et al., 2015).

Carbon/iron composites were prepared from waste cation exchange resin by NaOH activation and were used to remove diethyl phthalate, BPA and malachite green from aqueous solutions. Activated carbon/iron composites (800ACS-1), synthesized from waste cation exchange resins at 800°C also exhibited high stability over a wide pH range of 4–11.5 (Shi et al., 2014).

Fly-ash composite was used for the removal of CPs and BPA (Pan J.M. et al., 2011). In another investigation, sepiolite (SEP) was modified by acid and heat treatment (AH-SEP), and organic treatment (OAH-SEP) followed by preparation of polyethersulfone (PES)/AH-SEP and PES/OAH-SEP hybrid particles using a liquid–liquid phase separation technique, and were used for the removal of ethidium bromide and BPA (Cheng et al., 2011). A cationic surfactant Dodecyl trimethyl ammonium chloride modified silica gel was used to remove MnCP (Raval and Desai, 2016). Tripathi et al. demonstrated high performance removal of BPA with adsorption capacity of 203mgg^{-1} using hollow carbon porous nanospheres (HCPNSs) by extending the Stöber method for the one pot preparation of core@two-polymeric-shells structured composite. The core was prepared by tetraethyl orthosilicate and two polymeric shells were prepared by phloroglucinol-terephthalaldehyde and cresol successively. The obtained composite was carbonized in N_2 flow to produce silica@carbon (Si@C) nanospheres(Tripathi P.K. et al., 2014).

1.2.2 Degradation of Nitrophenols

An innovative treatment method is required either for the removal of persistent pollutant or for the conversion to a harmless product. However, some organic compounds, which

are very challenging for biochemical degradation, can be converted into degradable intermediate organics or completely oxidized to H_2O and CO_2 (Zhu F.-L. et al., 2016). Nitrophenols for instance are recalcitrant to biodegradation (Ju and Parales, 2010).

1.2.2.1 Advanced Oxidation Processes

Advanced Oxidation Processes (AOPs), are considered the most important technologies for environmental remediation of natural and wastewaters containing recalcitrant compounds, through the production of reactive oxygen species (ROS) (Boreen et al., 2003; Gogate and Pandit, 2004). AOPs are based on the in-situ generation of highly reactive hydroxyl radicals (·OH) in mild conditions (Li X. et al., 2014; Wang N. et al., 2014). Once formed, ·OH ubiquitously oxidizes and degrades efficiently the water contaminants to non-toxic, low molecular weight compounds (Mousset et al., 2016; Mvula and von Sonntag, 2003). The use of primary oxidants (e.g. ozone, hydrogen peroxide, dioxygen) (Fernandez-Castro et al., 2015; Martin-Martinez et al., 2016) energy sources such as ultraviolet light (Ge et al., 2009; Li Y. et al., 2012) or catalysts such as titanium dioxide, iron (III), cobalt(II), potassium permanganate, hybrids of TiO_2 catalysts with graphene, Fe_2O_3 (Chaliha et al., 2008; Darabdhara et al., 2016; Das et al., 2016; Elbanna et al., 2016; Jiang et al., 2015; Li H. et al., 2013; Malengreaux et al., 2014; Pera-Titus et al., 2004; Tasseroul et al., 2012; Thompson and Yates, 2006; Wang T.C. et al., 2011; Wang et al., 2017) are the current pathways of generating hydroxyl radicals and other ROS such as superoxide radical ($O_2^{\cdot-}$), peroxyl radical, singlet oxygen and sulfate radicals (Gogate and Pandit, 2004; Pang and Lei, 2016; Zhou et al., 2015). Hence AOPs, including photocatalysis (Araña et al., 2007; Feng et al., 2013); ozonation (Kuosa et al., 2009; Ma et al., 2014; Shahidi et al., 2015), Fenton (Pradhan and Gogate, 2010; Wang N. et al., 2014) and electro-Fenton (Zhou et al., 2014, 2013) have frequently been used to degrade 4-NP. Despite the advantage of removing organic compounds in aqueous phase AOPs hold several drawbacks which prevent their full-scale implementation; most prominently, AOPs costs are very high, requiring continuous inputs of expensive chemicals in order to maintain the operability of the oxidative system (Fernandez-Castro et al., 2015). Many kinds of microwave catalysts have been explored for the degradation

of phenolic pollutants in waste water (Bi et al., 2007; Bo et al., 2008; Jibril et al., 2013; Liu et al., 2010; Yin et al., 2016).

Photocatalysis

Photocatalysis, as a green technology, has attracted extensive interest in pollutant degradation (Chang et al., 2015; Li H. et al., 2015; Park H. et al., 2016; Ye et al., 2014). TiO_2 has been recognized as the most efficient photocatalyst for the degradation of organic pollutants in water and wastewater. The degradation of organic pollutants in water and wastewater (Liu C. et al., 2016; Pei et al., 2015). However, the band gap of TiO_2 which is larger than 3.0 eV limits its photoactivity to the UV region, making TiO_2 an inefficient photocatalyst for solar light utilization. The band gap is reduced by doping impurities into TiO_2 lattice (Nishiyama et al., 2016).

Narendra Singh et al. fabricated mesoporous, hollow TiO_2 nanofibers by a coaxial electrospinning technique for the photocatalytic degradation of 4-NP, a well-known model water pollutant. The hollow nanofibers were sensitized by cadmium sulphide (CdS) quantum dots (QDs) through successive ion layer adsorption and reaction (SILAR) method (Singh et al., 2016).

Degradation of phenol, 4-CP, and 4-NP (separate or mixed) and their intermediates was performed using TiO_2/reduced graphene oxide nanocomposite photocatalyst using visible light as photoexcitation source (UV and Xenon lamps) in the presence and absence of H_2O_2 by Al-Kandari et al. (Al-Kandari et al., 2016).

CuO nanoneedles were investigated for photocatalytic decomposition of rhodamine B, methylene blue and 4-NP by Momeni et al. (Momeni et al., 2016). Gangarajula et al., performed the photodegradation of 4-NP using TiO_2-Sr hydroxyapatite (Gangarajula et al., 2015). Typical dyes and aromatic compounds, including rhodamine-B, methyl orange, methyl blue, phenol and 4-NP, as well as bacterial (*E. coli*) cells were effectively degraded/inactivated in the presence of m-Bi_2O_4 which functioned as visible-light-driven photocatalyst (Wang et al., 2015).

Conventional treatment approaches have some major shortcomings. The slow degradation rate, limited degradation efficiency, secondary pollution, high costs and strict

operating conditions remarkably limit their wide applications in large scale (Huang et al., 2012; Liu Z. et al., 2012; Roy et al., 2013).

In this pursuit, **nanocatalysis** is an attractive research area in which metal nanoparticles are used as catalysts for a wide range of oxidation and reduction chemical reactions to convert nitrophenols to useful products. Metal and metal oxide nanoparticles based catalyses have attracted great attention in recent past because of the specific catalytic properties, large surface area-to-volume ratio, and other fascinating chemical and physical properties (Sun et al., 2013). Zinc oxide nanoparticles have been synthesized by a sol-gel microwave assisted method using either ethylene glycol or poly ethylene glycol ethanolic solution as dispersing media and have been used as photocatalysts for the catalytic degradation of 4-NP (Manuchehri et al., 2016). Fe-bearing attapulgite was used as catalyst, MW absorber, and Cd stabilizer in MW system and to oxidise 4- NP for remediation of 4-NP-Cd contaminated soil by Wang et al. (Wang X. et al., 2016). The 4-NP oxidation proceeds by initial aromatic hydroxyl oxidative attack followed by oxidative fragmentation, with release of nitrates 4-nitrocatechol, hydroquinone, benzoquinone and carboxylic acids thus increasing the biodegradability of nitrophenols (Martín-Hernández et al., 2012).

1.2.2.2 Hydrogenate reduction method

Recently, hydrogenate reduction method has received increased attention for remediation of NPs, as the generated aminophenol can serve as an important intermediate for cosmetic products, analgesics and antipyretics (Sun et al., 2014); which is a feasible way to convert pollutants such as nitrophenol to renewable resource. Further, aminophenol is relatively less toxic and easier to be removed and mineralized compared to nitroaromatic compounds. Wu and Yang reported the catalytic activity of Au nps for the reduction of 4-NP to 4-AP by $NaBH_4$ (Wu and Yang, 2011). Microwave assisted synthesized Cu nps have been used effectively as reduction catalysts for the degradation of organic pollutants methylene blue,methyl red,methyl orange, eosin yellow, 2-NP and 3-nitrophenol (3-NP) by Sreeju and co-workers (Sreeju. et al., 2016). The bimetallic nanoparticles can be alloy or core–shell and can be tuned both by preparation conditions as well as constituent metals' properties. The enhancement of catalytic activity of

bimetallic structures compared to monometallic nps could be mostly related to: (1) the small diameter and (2) electron transfer between metals in the bimetallic structures, resulting in catalytic active sites (Zaleska-Medynska et al., 2016). For instance, Au-Ag alloyed nps with different morphologies and sub-nanoscale crystalline structures were synthesized by adjusting the size of the sacrificial Agnps used in the galvanic replacement reaction. The atomic crystalline structure of Au-Ag bimetallic nps was found to play a much more significant role than morphology in determining the catalytic ability of the nanostructures for the catalytic reduction of 4-NP by $NaBH_4$ to 4-AP. The Au-Ag alloyed hollow and porous nanostructures with better surface crystalline alloying micro-structures and open morphologies presented much higher catalytic reaction rates and better cyclic usage efficiencies. This was attributed to the effective alloyed crystalline microstructure surfaces which provided better dispersions of the active Au atoms contributing to the higher catalytic activities of the structures (Liu R. et al., 2016). Gold was recovered as Au nps by Zhu et al. from industrial effluent streams by Shewanella haliotis with sodium lactate as electron donor and the Aunps were used for catalytic degradation of 4-NP (Zhu N. et al., 2016). The high catalytic properties of such materials highly depend on the size and shape of nanoparticles.

However, the very small size of the nps also complicates their separation and recovery after completion of the reaction. Another problem with pristine catalysts is their catalytic efficiency and aggregation. To prevent the aggregation, the nps are stabilized with various agents, such as charged molecules (Dong et al., 2009), polyelectrolytes (Kidambi et al., 2004) and dendrimers (Rajesh et al., 2014). These stabilizing agents not only protect the nps but also ensure their dispersion in polar and non-polar solvents. Wenbo Lu et al. synthesized Au nps decorated GO nanosheets using polyoxyethylene sorbitol anhydride monolaurate (TWEEN 20) as a stabilizing agent for GO as well as a reducing and immobilizing agent for Aunps and used the sheets for catalytic reduction of 4-NP (Lu et al., 2011). Gallic acid was used as both reducing and stabilizing agent to synthesize Au and Ag nps.

Raveendran et al., have initiated the green synthesis of nps (Raveendran et al., 2003). Since then biofabrication of nps is of interest (Ajitha et al., 2016; Khan et al., 2016; Park

J. et al., 2016).Plant extracts contained abundant chemical constituents, such as phenols, reducing sugars, ascorbic acids and others, which were responsible for the bioreduction of metal ions and the stabilization of the nps (Wei et al., 2016). Microbes mediated nps have the natural propensity for detoxification of metallic ions, where biomolecules secreted by the biomass extracellularly or intracellularly, can act as both reducing as well as capping agents during the reaction process finally forming stable nps, which are more biocompatible (Lukman et al., 2011).

Biogenically synthesised Au nps using the yeast cells of Magnusiomyces ingens were investigated for the catalytic reduction of NPs (i.e. 2-NP, 3-NP and 4-NP) to AP by Zhang et al. (Zhang X. et al., 2016). Areca catechu nut derived Au nps were used by Rajan et al. for degradation of Methylene blue, Methyl orange, Eosin yellow and 4-NP (Rajan et al., 2015).

Further hollow porous Aunps (Guo M. et al., 2016), Agnps derived from plant extract of Piper betle (Ankamwar et al., 2016) have been investigated for catalytic reduction of 4-NP. Mono-disperse Au nps with sizes between 2.3 and 23.1 nm were synthesized by Lin et al. with the assistance of a natural wood material as a reductant/support that exhibited high catalytic activity for the reduction of 4-NP as a model reaction (Lin et al., 2016) Nazirov et al. utilized a green method for Au (III) reduction using biocompatible nontoxic N-(4-imidazolyl) methylchitosan as both reducing and stabilizing agent for complete conversion of 4-NP to 4-AP (Nazirov et al., 2016).

Green synthesis of Aunps was done by a newly isolated strain Trichosporon montevideense for catalytic hydrogenation of nitroaromatics (Shen et al., 2016).

Magnetic core–shell carbon dot@MFe_2O_4 (C-dot@MFe_2O_4) (M= Mn, Zn and Cu) hybrid materials have been investigated for catalytic degradation of 4-NP and for the degradation of other organic dyes, such as methylene blue and rhodamine B (Guo Y. et al., 2016). Moreover, the presence of magnetic component not only endows the catalyst with magnetically separable ability from a suspension system, but also significantly lowers the cost (Xia et al., 2017).

Immobilization of nps is also reported on various substrates to prevent aggregation such as on alumina (Ivanova et al., 2006), silica spheres (Qiu et al., 2011), zeolites (Chen et

al., 2005), SBA-15 (Zhu et al., 2013), graphene (Hu J. et al., 2014), polymeric substrates (Zhou et al., 2016), mesoporous silica (Mehta et al., 2016). An Aunp–rGO/QCS (Quaternized chitosan) /cellulose paper was used as catalytic substrate material (Ling et al., 2015). Qi Wang et al. observed that rGO/ Aunps/Tyr (tyrosine) nanocomposite exhibited good catalytic activity toward 4-NP reduction (Wang Q. et al., 2016). Reports are available, which suggest the coupling of separation with catalysis in membrane reactors where in situ separation of nps can be achieved (Chen et al., 2009). Separation can be achieved by immobilization of the nps on the surface and inside the pores of the membranes by suitable chemical modification using different crosslinking or coupling agents to generate sufficient functional groups or by grafting of polymer brushes (Clodt et al., 2013; Li S. et al., 2013; Subair et al., 2016; Tripathi B.P. et al., 2014). Two-dimensional graphene/ SnO_2 composite nanosheets hybrid nanostructures consisting of 5 nm Pt nps supported on the both sides of hybrid nanostructures was synthesized and used for the catalytic reduction of 4-NP by Zhu et al. (Zhu et al., 2011).

Superporous **cryogels** of poly(2-hydroxy ethyl methacrylate) (Sahiner et al., 2015), poly(3-sulfopropyl methacrylate), poly-(acrylic acid) (Sahiner et al., 2016), poly(4-vinylpyridine) (Sahiner and Yildiz, 2014), poly(2-acrylamido-2-methyl-1-propansulfonic acid) (Sahiner and Seven, 2014) and their templated metal np composites (Co, Ni, Cu, and Fe) were used in hydrogen generation from the hydrolysis of $NaBH_4$ and hydrogenation of 4-NP. Betaine type microgel based on poly-2-[(methacryloyloxy) ethyl] dimethyl (3-sulfopropyl) ammonium hydroxide, was used as template for the in situ synthesis of Ni nps and as catalysts for hydrogenation of nitrogroup containing substrates 4-NP, 2-NP and 4-nitroaniline (Ajmal et al., 2015). Amphoteric cryogel with immobilized Aunps was tested as flow-through catalytic reactor in reduction of 4-NP by $NaBH_4$ (Tatykhanova et al., 2016). Hai Bang Ly et al. reported the preparation of doubly porous poly (2-hydroxyethyl methacrylate)-based materials, and their subsequent functionalization with different organic compounds of interest, namely cysteamine, ethylenediamine, and propargylamine and utilization as Aunps support for catalytic reduction of 4-NP (Ly et al., 2016). Amphiphilic hyperbranched polymers with polyethylene glycol chains and low-molecular-weight polyethylenimine conjugated to

commercial aliphatic hyperbranched polyesters (Boltorn Hx) were used to stabilize Aunps at the interlayer between the core and the shell for catalytic reduction of 4-NP (Dai et al., 2016a).

A novel 3D Ag (I)-MOF with $ThSi_2$ (10^3-b) topology directed by sodium caprylate was used by Wu et al. towards the degradation of 2-NP, 3-NP and 4-NP in aqueous solution(Wu X.-Q. et al., 2016). Cu/Chitosan- cellulose microfiber mat was used as a reduction catalyst for the degradation of nitro-aromatic compounds as well as an organic cresyl blue dye by Haider et al. (Haider et al., 2016). Au and Agnps/ peptide nanofibers synthesized by Xu et al. exhibited excellent catalytic activity towards the reduction of 4-NP, with turnover frequency (TOF) values of 720, 188, and 96 h^{-1}, respectively (Xu W. et al., 2016). Grass-like double-layer ZnO micro-nanostructures confined in microchannels fabricated via fluid construction methods, with uniformly distributed catalytic Pt micro-nanostructures were used by Zhang et al. for the continuous reduction reaction of 2-NP and degradation of methylene blue (Zhang W. et al., 2016).

Cellulose nanocrystal-supported Au nps was reported to exhibit catalytic activities for the reduction of 4-NP by sodium borohydride (Yan et al., 2016). Thiol-functionalized hierarchically porous materials prepared by combination of hyper-cross-linking and molecular templating of core-shell bottle brush copolymers were investigated for their catalytic performance on reduction of 4-NP (Xu Y. et al., 2016). Ansar et al. investigated thiolated polyethylene glycol as a stabilizing ligand for Au nps during catalytic reduction of 4-NP (Ansar and Kitchens, 2016). Cucurbit [7] uril-protected Au nps were used for catalytic degradation of nitrofurantoin and 4-NP (Blanco et al., 2016). Hierarchically porous nitrogen-doped MOFs embedded with cobalt nps (Co@NC) were prepared using a well-defined rhombic dodecahedral cobalt-based zeolitic imidazolate framework-67 (ZIF-67-Co) as an effective precursor and template. The resulting Co@NC exhibited remarkable catalytic activity and excellent durability for the reduction of 4-NP to 4-AP by $NaBH_4$ in an aqueous solution (Li X. et al., 2016). Graphene composite, graphene/Fe_3O_4/NiO nanocomposites (Zhao G. et al., 2015) were used for the catalytic degradation of organic pollutants such as 4-NP and Rhodamine B. rGO was synthesized via functionalization with metals and metal oxides using sodium sorbate as a stabilizer.

Single (Ag, Cu_2O, and Fe_3O_4) and bimetallic (Ag-Cu_2O, Ag-Fe_3O_4 and Cu_2O-Fe_3O_4) nps were incorporated on the surface of rGO and were applied for inactivation of E. coli, catalytic degradation of 4-NP as well as for adsorption of As (V) (Dubey et al., 2015).

Aunps anchored on oxidized OMC was used for catalytic reduction of 4-NP to 4-AP rapidly in mild condition by Gao et al. (Gao W. et al., 2016).

1.3 Methods of Analysis

A number of analytical techniques have been described in the literature for the determination of nitrophenols which include gas chromatography (GC) (Tesarova, E.; Sykorra, D.; Voznakova, 1995), gas chromatography/mass spectrometry (GC/MS) (Auroux et al., 2002), high performance liquid chromatography (HPLC) (Yamauchi et al., 2004), capillary electrophoresis (Gao and Ren, 2010; Guo et al., 2004; Niazi and Yazdanipour, 2007; Nistor et al., 2001; Zhang et al., 2012), flow-injection analysis (Manera et al., 2007), spectroscopic methods (Niazi & Yazdanipour, 2007; L. Gao & Ren, 2010) and enzyme-linked immunosorbent assay (Tingry et al., 2006). Though highly sensitive, these methods require expensive instruments, a lot of expertise during operation and are not easy to be deployed in the field due to their bulky size. Furthermore, it is highly desirable in aqueous media for the field selective detection of nitroaromatic compounds present in soil and ground water.

However, the sample matrix is usually very complex and the ordinary detection method would not be universally applicable. Each analytical technique for detection and sensing of NPs has issues regarding pretreatment requirements, compound recoveries, detection limits, and compound specificities. Because of the presence of NPs at trace levels in the various matrices, especially water samples, the analytical methods with effective sample preparation and extraction, and trace-level detection are required (Feng et al., 2009; Moradi et al., 2013).

Electrochemical methods have shown better potential for 4-NP determination because of the advantages of low cost instrumentation, easy operation, good sensitivity, and short run times with simple sample pre-treatment and in situ detection (Xu et al., 2013). However, the electrochemical detection of 4-NP at bare electrodes offers less sensitivity and suffers from high overpotential and interference problems (Yang et al., 2011).

Therefore, chemically modified electrodes are widely employed to overcome these disadvantages, leading to the development of highly efficient electrochemical sensors for the determination of 4-NP (Lima et al., 2014; Gao J. et al., 2016; Ikhsan et al., 2016; Yin et al., 2010). Yang and co-workers have developed a rapid and sensitive electrochemical sensor using coupled carboxyl- MWCNT and CD edge-functionalized graphene composite towards trace detection of 4-AP, 4-CP and 4-NP (Yang et al., 2015). Many modified electrodes based on CNTs (Luo et al., 2008; Yang, 2004), metal nps (Casella and Contursi, 2007; Chu et al., 2011; de Lima et al., 2014; Maduraiveeran and Ramaraj, 2009; Tang et al., 2015; Zhang et al., 2011), and ionic liquids (Sun et al., 2008) have been reported for the determination of 4-NP. Ikhsan et al. developed an electrochemical sensing platform comprising of rGO-Ag nanocomposite for the detection of 4-NP (Ikhsan et al., 2016). A ZnO coated Glassy Carbon Electrode (GCE) was used by Bashami et al. for detection of 4-NP (Bashami et al., 2015). Ahmad at al. modified GCE using perovskite ($SrTiO_3$) and r-GO based nanocomposite (r-GO/$SrTiO_3$) for electrochemical detection of nitroaromatics (Ahmad et al., 2016). Cationic tetra-(N,N,N-trimethylaminoethoxy) phthalocyaninecobalt(II)/acid-treated CNTs multilayer films, were constructed on GCE by a facile, effective layer by layer (LBL) electrostatic assembly method for detection of 4-NP (Wu H. et al., 2016).

Luo et al. have fabricated MIPs on the surface of graphene sheet wherein vinyl group functionalized graphene was first prepared by immobilizing 4-vinylcarbazole onto the surface of graphene via π-π interactions. The subsequent grafting copolymerization of methacrylic acid and ethylene glycol dimethacrylate in the presence of 4-NP (template molecule) was carried out, leading to the formation of graphene MIPs composite. The electrochemical behavior of graphene-MIPs sensor for 4-NP were investigated by cyclic voltammetry and differential pulse voltammetry methods (Luo et al., 2015).

Complicated synthetic steps, sample pretreatment and assay processes are involved in electrochemical techniques, which limit their applications for analysis of NPs. Consequently, it is desirable to develop a rapid, relatively simple and inexpensive method for trace analysis of nitrophenols.

Overcoming these limitations, **chemical sensors** have emerged as simple, rapid, cost-effective and portable alternative tool for analyzing environmental samples. Spectrophotometric (Üzer et al., 2004) and fluorescence (Wang et al., 2006; Zarei and Ghazanchayi, 2016), techniques for nitroaromatics have evolved.

A great number of fluorescence-based materials such as conjugated polymers (Germain and Knapp, 2009; Hu Z. et al., 2014; McQuade et al., 2000; Salinas et al., 2012; Sawa, 2008; Thomas et al., 2007), nanoparticles (Fan et al., 2016; Guo Y. et al., 2016; Jiang et al., 2008; Saini et al., 2016; Yue et al., 2016) and microporous metal-organic frameworks (MOFs) (Bo et al., 2014; Cao et al., 2015; Guo et al., 2014; Liu et al., 2015; Qin et al., 2016; Singha et al., 2015a, 2015b, 2014; Song et al., 2014; Wu et al., 2015; Zhang Q. et al., 2016; Zhao S.-N. et al., 2015) have been applied towards the detection of nitroaromatic compounds. Many nanosensors have been investigated to determine TNP (Bai et al., 2015; Enkin et al., 2014; Liu et al., 2013; Shen et al., 2012; Zhang L. et al., 2016). A variety of luminescent materials containing nanoparticles, organics and biological molecules have been employed for detectionof 4-NP (Nistor et al., 2001; Paliwal et al., 2007; Yang et al., 2014). However, the wide- spread uses of these materials are stil limited due to their low stability, toxicity and sensitivity.

Zinc and cadmium **coordination polymers** with terephthalate and dipyridylamide ligands via luminescence quenching have been applied in nitroaromatic-sensing (Wudkewych and LaDuca, 2016).

A luminescent Zn (II)-organic framework, $[Zn(PIA)]_n.2nDMA$ [where H_2PIA is 5-(pyridin-4-yl)isophthalic acid and DMA is N,N-dimethylacetamide], was solvothermally synthesized and was reported to be a potential luminescent sensory material for 4-NP (Zhang J.R. et al., 2016). The synergy of accessible porosity within microporous MOFs and fluorescence provided them with the ability of converting the host-guest interactions to detectable fluorescent changes, which further makes them potential candidates for sensing applications.

The solvothermal reaction of $Gd(NO_3)_3.6H_2O$ with the multidentate π-conjugated ligand H_4L(5,5'- (pyridine-2,5-diyl)-isophthalic acid) having a lewis basic pyridyl site resulted in the synthesis of a microporous luminescent Gd-MOF

$[Gd_6(L)_3(HL)_2(H_2O)_{10}.18H_2O.x$(solvent) that demonstrated high sensitivity and selectivity towards trace determination of 2-, 3, and 4-NPs and Fe^{3+} ions with good linearity (Tan et al., 2016).

A ratiometric fluorescent **molecularly imprinted** sensor has been constructed for highly sensitive and selective detection of 4-NP using carbon dots (CDs) as the target sensitive fluorophore and YVO_4: Eu^{3+} nps as the reference fluorophore (Li W. et al., 2016).

In recent years, numerous **graphene-derived carbon materials** have been synthesized and applied as molecular support and carrier platforms (Bao Q. et al., 2011), sensors (Liu R. et al., 2012; Shao et al., 2010; Song and Zhu, 2013), aptamers (Hu et al., 2012; Wang et al., 2010) and electrodes (Park et al., 2012; Wang et al., 2008).

Graphene quantum dots (GQDs) are single carbon layer thick luminescent nano-materials; the sheets are generally smaller than 100 nm (Zhuo et al., 2012) with oxygen-containing functional groups at the edges. Such nano-materials have unusual optical and electronic properties partly arising from their quantum confinement and zigzag edge effects (Bao L. et al., 2011; Jin et al., 2013; Yan et al., 2013). Other useful properties include: chemical inertness, strong fluorescence, high photo-stability, and low toxicity (Güttinger et al., 2012; Li L.-L. et al., 2012; Shen et al., 2012; Zhu et al., 2012).

Min Bai's group functionalized β-cysteamine on the surface of fluorescent QDs, which promoted the electron transfer from amine groups to nitromoieties, resulting in fluorescence quenching of QDs by TNP (Bai et al., 2015).

Ag nanoclusters templated by hyperbranched polyethyleneimine with different terminal groups and molecular weights were developed as special optical sensors for detecting 4-NP (Qu et al., 2017).

The brief literature review reveals that palm shell based activated carbons have not been used for removal of organic pollutants nor as supports for nanoparticles. The uses of agrowaste derive carbon as catalyst support has been hardly reported. Though CDs and GQDs appear to be simple and attractive as sensors, to the best of our knowledge reports of carbon dots and graphene as sensors of nitroaromatic compounds in aqueous media are relatively rare, especially for sensing 4-NP, DNP and TNP.

It was thus felt that investigations with following objectives would be useful

- ❖ Development of activated carbons (ACs) which can be utilized both as adsorbents and porous support.
- ❖ Characterization and evaluation of the adsorption potential of ACs for the removal of selected model organic pollutants like Bisphenol A, 4-Chlorophenol, 4-Nitrophenol, 2,4-Dinitrophenol, 2,4,6-Trinitrophenol and Monocrotophos.
- ❖ Use of the prepared activated carbons as supports for nano silver.
- ❖ Application of the nano silver loaded activated carbons as catalysts for the model reduction of nitrophenols to aminophenols.
- ❖ Preparation and characterisation of novel carbon based materials like graphene and carbon quantum dots.
- ❖ Application of carbon quantum dots and Graphene quantum dots as sensors for phenols and pesticides like monocrotophos.

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