

Hazardous Effects of Fluoride and Its Removal Techniques: A Review

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ABSTRACT

Fluorine (F) is highly electronegative in nature and reactive non-metal element. It is rarely found in pure form. It is found in combined form with other elements except oxygen and noble gases. An inorganic monoatomic F anion is called fluoride (F^-). Although F^- is the reason for healthy teeth and bones, excess F^- can be detrimental to human and animal health. The rising prevalence of F^- -related problems with human, animal, and plant health is mostly due to natural geological sources and expanding industrialization. The main source of daily F^- consumption in human is potable water. Dental and skeletal fluorosis is caused by F^- concentrations that are above the recommended limits. Some other effects include programmed cell-death, endocytosis, metabolic disorders, osteoporosis, joint pain, weakness of muscles, non-ulcer dyspepsia, polydipsia, anaemia, etc. For human safety and control, removal of F^- is necessary. Therefore, it is important to gather knowledge on the fundamental problems related to F^- contamination, such as origin of F^- disclosure, negative impact on health, along with the viability of F^- removal techniques. This review provides a comprehensive summary of the F^- removal techniques in recent use along with their benefits and drawbacks.

Key Words: Adsorption, De-fluoridation, Fluorosis, Inverse fluidization, Wastewater.

1. INTRODUCTION

One of the most basic necessities of human being is water and the access to drinking or potable water is necessary for retaining good health [1]. The rise in water contamination is due to large human population, urbanization, residential complexes, agricultural practices, and due to other environmental modifications [2]. Excess concentrations of nitrate, arsenic, and F^- ions are among the chemical contaminants that have been proven to have negative effects on the health of living beings. The origin of the F^- contamination might be either anthropogenic or geogenic. Anthropogenic F^- contamination is when there is a large increase in F^- contamination as a result of human activity, while geogenic F^- contamination is when there is a major increase in F^- contamination as a result of natural processes [3]. Sedimentary and igneous-type rocks contain minerals such as sellaite (MgF_2), fluorapatite ($Ca_5(PO_4)_3F$), cryolite (Na_2AlF_6), and fluorite (CaF_2) which enter into potable water naturally. F^- in potable water may have positive or negative effects on human being depending on the concentration and length of continuous intake. Due to particular geographical, economical, and financial disadvantages, some places of the World lack access to potable water [1]. Regions with poor water quality often leads to grave social and health problems. Because of this, providing potable water is considered as a prime concern in many countries. Developed and underdeveloped countries are not able to meet the potable water criteria as per the guidelines. The World Health Organization (WHO) reported that, nearly 783 million people are deprived of improved water sources. It is predicted that half of the World population would face a shortage of potable water by 2025 [4]. Above 200 million individuals rely on water of high F^- concentrations which is above 1.5 mg/L. Concentrations above these limits have been associated with dental and skeletal fluorosis. In mild cases tooth mottling occurs, but bone fragility and neurological problem takes place in severe cases.

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Osteoporosis, kidney disease, cancer, brain malfunction, and bladder cancer may happen. Infertility, Alzheimer syndrome, gastro-intestinal irritation, thyroid disorder, etc. can occur due to high F^- intake [5]. As it shows several adverse effects on human

health, there is a need to develop, on urgent basis, a reliable technology that can effectively remove excess F^- from potable water. Defluoridation is the ideal solution to this problem. Different techniques are currently in use for removing F^- from water/wastewater. The aim of this review article is to briefly describe range of concerns associated with F^- contamination such as sources, its harmful effects, and F^- removal techniques with regard to their applicability.

2. SOURCES OF FLUORIDE

F^- is emitted to the atmosphere both in gaseous as well as in particulate forms [6]. Further, exposure of F^- to the environment can be grouped into natural and anthropogenic sources and industrial wastes [7,8].

2.1 Natural Sources

2.1.1 Water

Especially in urban areas, F^- can be found in rainwater, groundwater, freshwater, saltwater, and other sources [9-11]. The average F^- concentration in seawater is 1 mg/L while the value fluctuates for freshwater. Its average concentration in river water and groundwater are 1.5 and 1.0–35.0 mg/L respectively [12]. Almost 75 to 90% of consumed F^- is absorbed and under acidic stomach condition, hydrogen fluoride is formed inside stomach. The existence of nearby F^- containing minerals affects the quality of ground water (well water) to a greater extent. For example, some regions of Canada and China have elevated F^- levels of 4.3 and 13 mg/L respectively [13]. F^- rich minerals are fluorospar, cryolite, topaz, fluorite, phosphorite, theorapatite, and fluorapatite [14,15]. It enters the water through weathering.

2.1.2 Plants

Small amounts of F^- are found in agricultural foods like vegetables and food grains [16]. These foods adsorb F^- rapidly from soil. Use of different type of fertilizers and pesticides influence the quality of foods. Also it depends on the water used for the production of foodstuffs. Soils with high F^- concentration are mostly found in industrial areas. Tea is another major source of F^- [17,18]. The majority cases of fluorosis are found in some places of China because of higher consumption of tea [18]. Matured tea leaves from the same plant can have up to 10 times higher F^- levels than young tea leaves [19]. The concentration of F^- in different foodstuffs is given in Table 1 [20].

2.1.3 Soil

In normal soil, total F^- level varies between 150 to 400 mg/kg while in heavy clay soil, it is above 1000 mg/kg [21-25]. Use of phosphatic fertilizers increases the F^- contamination of the soil with the level of F^- at around 1 to 1.5% [26]. F^- contaminated soil have toxic effects. These are mixed with underground water due to leaching from the soil [1].

2.1.4 Volcanic Activities

The global animal and plant kingdom is impacted by volcanic eruptions. Ashes from volcanic eruptions have high level of F^- which frequently contaminates the geochemical cycle. F^- has been released as hydrogen fluoride from the volcanic outburst. F^- that has erupted may cover a larger land area and may continue to be in land for decades. Many domestic and wild animals are mostly affected by decaying and leaching of volcanos [7,27,28].

2.2 Anthropogenic Sources

Industrialization and motorization are the cause of high F^- exposure to the environment. Many anthropogenic sources are F^- containing pesticides, fluoridated potable water, and dental products. Some food based F^- sources are tea based drinks, processed foods, and beverages [29]. Some other F^- sources are industrial solvents, local anaesthetics, refrigerants, fire extinguishers, and glass chillers [30]. Contamination also occurs due to air sources. In unpolluted areas, the average F^- concentration is lower than $0.1 \mu\text{g}/\text{m}^3$. F^- level is around 2 to $3 \mu\text{g}/\text{m}^3$ in industrial areas [27].

The primary source of F^- known to cause endemic fluorosis in many countries is coal burning for domestic use [6,31]. Cattle, buffaloes, sheep, goats, camels, and other animals in the livestock industry have died as a result of industrial release of F^- rich gases and effluents into the environment [32-35].

2.3 Industrial Wastes

In industries, F^- salts and hydrofluoric acid plays a significant role. In the past it was mined to be used in aluminium smelting. But currently it is derived from hydrogen fluoride. For the separation of slag in steel-making, usually fluorite is utilized on a large scale. Fluorocarbons are produced by the help of hydrofluoric acid and hydrogen fluoride. Hydrofluoric acid is used to dissolve glass [36]. F^- is also used in casting technologies, abrasive industry, and automotive industry (for braking system). F^- wastes generated from these industries in gaseous, liquid, and solid forms pollute the environment.

Table 1. Fluoride levels of different food items

Food items	Fluoride concentration (ppm)	Food items	Fluoride concentration (ppm)
Buffalo milk	3.32-6.85	Apple	1.05-2.20
Wheat	0.51-14.03	Red gram	2.34-4.84
Cow milk	1.73-6.87	Cabbage	4.25-11.30
Rice	0.51-5.52	Peas	10.77
Soybean	4.0	Spinach	9.87-29.15
Bajra	2.76-3.84	Fermented milk items	1.76-93.68
Maize	5.6	Green tea leaf	72.62-89.02
Grape	0.84-1.74	Bengal gram	3.84-4.84

3. APPLICATIONS

F^- is used for dental caries, for water fluoridation, as biological reagents, and also used in many different industries. Some of these are mentioned here.

3.1 Cavity Prevention

F^- is used to inhibit tooth decay. It is used to fluoridate water and used in a variety of oral hygiene products [37,38]. Several bacteria in the mouth contribute to tooth decay. Carbohydrate and sugars help these bacteria to create acid. Tooth decay process is stopped by F^- and it strengthens the tooth enamel. F^- causes the removal of minerals from the surface of the tooth. F^- assists in remineralizing the surfaces of tooth and reduces cavity formation [38].

3.2 Water Fluoridation

As a dental caries prevention measure, in 1945, several areas of the United States (US) started fluoridating their water supply [39]. Approximately three out of four Americans currently have access to fluoridated potable water. Distinct forms of F^- such as sodium fluorosilicate, fluorosilicic acid, and sodium fluoride are added to various water systems [40]. In the US, F^- is present in several sources of natural potable water, though the amounts vary from region to region. The US Public Health Service advisory for supply water is to have 0.7-1.2 mg/L of F^- for the prevention of tooth decay [40-42]. Permitted value of F^- depends on the annual average air temperature of the location. 2.4 mg/L is the permissible level for bottled water without additional F^- at very lower temperatures. The US Food and Drug Administration recommends that manufacturers should keep their F^- value within 0.7 mg/L [40]. But F^- value of 1.5 mg/L is decided by the WHO [13].

3.3 Biological Reagent

In biological test processing, F^- salts are frequently employed to block the function of protein tyrosine phosphatase [43]. F^- tries to copy the nucleophilic hydroxide ion. These ions are present at active site of enzymes [44]. Aluminum and beryllium fluorides act as phosphatase inhibitors. These two compounds have similar structure to phosphate group. Also they can function in a manner similar to the transition state of the reaction [45,46].

4. HAZARDOUS EFFECTS OF FLUORIDE

4.1 Health Impact of Fluoride on Humans

Due to higher F^- concentration, different kinds of diseases occur in human body. Different diseases occurring with their F^- concentration is presented in Table 2 [15].

4.1.1 Dental Fluorosis

The main component of tooth enamel is crystalline hydroxyapatite. Under normal conditions, in calciferous tissue enamel, apatite crystal lattices are incorporated with F^- ions during its formation. The ion of hydroxyl group is replaced by F^- because stability of fluorapatite is higher than hydroxyapatite [47]. In dental fluorosis, white spots on the surface of tooth enamel occur in mild cases. In severe cases, yellowish brown to black stains and extensive tooth pitting takes place. At the age of 8 to 10 years, dental fluorosis is highly visible which is based on the quantity of F^- consumption [48]. If the teeth have developed to their fullest extent before the excessive F^- exposure, the effects of dental fluorosis may not be immediately noticeable. So at adult age, if there is no symptom of dental fluorosis that does not confirm that their F^- intake is in safe range [15]. Nearly half of the consumed F^- is eliminated from human body by sweat, urine, and faeces [47].

4.1.2 Skeletal Fluorosis

Children and adults both suffer from skeletal fluorosis which is not easily detected unless an advanced stage is reached. F^- generally gets deposited in the joints resulting into difficulty in walking. At the initial stages limb tingling, muscle weakness, or stiffness in back may be experienced which may lead to osteoporosis at a later stage. Even it may cause rare bone cancer or may damage nervous system [49].

4.1.3 Other Ill-effects

High amount of F^- may also affect muscle fibre; urinary tract; respiratory, excretory, reproductive, gastrointestinal, or nervous systems; immunity; liver; kidney; red blood cells; or haemoglobin levels. It also destroys several enzymes [15]. Thyroid disorder, osteoporosis, brain damage, cancer as well as Alzheimer syndrome are the result of excess F^- intake [50]. Excess F^- levels have the potential to disrupt the synthesis of DNA as well as disrupt the metabolism of vitamins, carbohydrates, lipids, proteins, and minerals. Brain and pineal gland can also be damaged by high F^- consumption [51]. Abnormal behaviour in animals as well as reduction of human IQ are the effects of high F^- intake [52].

Table 2. Diseases occurring with their fluoride concentration

Diseases	Concentration of fluoride (mg/L)
Dental fluorosis	1.0-3.0
Stiffened and brittle bones	3.0-4.0
Crippling fluorosis	4.0-6.0 and above

4.2 Effect of Fluoride Contamination in Animals

High concentration of F^- also affect animals [7,53,54]. Food intake and body weight gain reduces due to F^- poisoning in animals [55,56]. Antioxidant enzymes such as catalase, glutathione peroxidase, and superoxide dismutase lose their activities. Easy penetration of F^- through the blood brain barrier resulted in neurodegeneration due to imbalances between oxidant and antioxidant levels [7]. Insecticide formulations contain F^- which if accidentally taken by animals

may cause intoxication [57]. Neurodegeneration is mostly attributed due to oxidative damage caused by excessive formation of reactive free radicals which is F^- induced [58].

F^- is absorbed directly from the water by aquatic animals like fish and invertebrates [51]. Marine bivalves are affected by F^- accumulation on the shell and meats [59]. Water temperature, exposure time, and F^- concentration affect the toxicity of F^- in fishes [60]. Despite the possibility of removal through excretory systems, it frequently accumulates in the fish bone tissue and in the exoskeleton of invertebrates. F^- also affects the enzymatic activities in fish [61].

Cows and other domestic animals show signs of skeletal and dental fluorosis. Some signs of nonskeletal fluorosis in these animals are excessive urine discharge, repeated abortions, intermittent diarrhoea, stillbirths, and colic [62].

4.3 Effect of Fluoride Contamination in Plants

F^- reduces quantity as well as quality of grains, fruits, vegetables, protein, fat, sugar, and forage [63]. F^- concentration, time of exposure, and types of algal species has great impact on the growth of algae which either accelerate or retard the growth of algae. Some algae can withstand high F^- concentration of 200 mg/L. Its ions hamper the cell division process governed by nucleotide and nucleic acid metabolism [64].

Varying levels of F^- are found in several foodstuffs such as in vegetables and fruits it is 0.1 to 0.4 mg/kg whereas in barley and rice it is up to 2 mg/kg [65]. Tea leaves has high F^- concentration of which 25 to 84% being dissolved in the liquor [66]. For agricultural land, source of F^- is phosphatic fertilizers [67]. *Freesia*, *Gladiolus*, and some other plant species show toxic indications at low F^- concentrations of 20 $\mu\text{g/g}$ dry weight. Whereas *Camellia* and Elderberry plant species show no toxic symptoms up to 3600 $\mu\text{g/g}$ dry weight of F^- . Toxic signs shown in plants are the reduction in chlorophyll production, leaf drop, and leaf necrosis [68]. Also tip burning and finally death of plants occurs in high F^- contaminated soil [69].

5. FLUORIDE CONTAMINATION IN INDIAN SCENARIO

Including India, 23 nations of the World is severely affected by F^- contaminated water. 177 districts of 20 Indian states face the problem of F^- contamination and among these Andhra Pradesh, Haryana, Rajasthan, Punjab, Uttar Pradesh, Tamil Nadu, and Gujarat are the most affected states. F^- concentration of half of Indian rivers is between 0.1 to 12 ppm [51]. In Rajasthan, people have no other option than using F^- contaminated water of up to 44 mg/L resulting into permanent deformities [70]. Fluorosis is a problem in some areas of the Anantapur district in Andhra Pradesh where the F^- concentration is between 0.56 and 5.8 mg/L [71]. A number of defluoridation techniques that have been developed, have higher operational and maintainance costs [72]. Developing countries stay away from adopting the expensive techniques [73]. One of the affordable and well-known defluoridation techniques used extensively in India is the Nalgonda technique. Coagulation and precipitation is the basic principle of Nalgonda technique [51,72]. In India, around 620 lakh people are facing the problem of fluorosis due to high F^- content of water [71]. In Odisha state, out of 30 districts, the potable water found in 27 districts is highly contaminated with F^- and 18 districts are affected by the problem of fluorosis [74].

6. FLUORIDE REMOVAL TECHNIQUES

Defluoridation refers to the lowering of F^- level to a recommended level of potable water and is considered to a reliable approach in the areas affected by fluorosis [75]. For the elimination of excess F^- from ground water in practical and affordable way, numerous techniques have been developed such as adsorption, reverse osmosis (RO), nanofiltration (NF), electrodialysis (ED), precipitation, ion-exchange, electrocoagulation (EC), freeze concentration, fluidized bed technology, biological treatment, nanotechnology, and hybrid techniques [76-80]. These techniques have their own strengths and weaknesses which are presented in Table 3.

Table 3. Strengths and weaknesses of different defluoridation techniques

Technique	Strengths	Weaknesses
Adsorption	<ul style="list-style-type: none"> • High performance • Cost effective • Ease of operation • Highly eco-friendly 	<ul style="list-style-type: none"> • Strong <i>pH</i> dependence • Influence of other ions • Regeneration requirement
Precipitation	<ul style="list-style-type: none"> • Low water wastage • Low energy requirement • No change in taste and odour 	<ul style="list-style-type: none"> • Heavy sludge formation • Produce high residual aluminium
NF	<ul style="list-style-type: none"> • Highly efficient F^- removal • Organic debris, bacteria, and suspended particles are all effectively rejected • Apply at wide <i>pH</i> range 	<ul style="list-style-type: none"> • Membrane fouling • Chemical resistance • Shorter membrane lifetime • Unsatisfactory elimination of some compounds
RO	<ul style="list-style-type: none"> • Longer lifetime of membrane • Less operative attention 	<ul style="list-style-type: none"> • Expensive technique • Limited membrane regeneration • High generation of acidic water
Dialysis and Electrodialysis	<ul style="list-style-type: none"> • High amount of water recovery • No membrane fouling • No requirement for specific solution storage 	<ul style="list-style-type: none"> • Unsuitable for rural areas • High voltage dependent • Intensive pre-treatment requirement • Frequent replacement of electrodes
Ion-exchange	<ul style="list-style-type: none"> • No change in taste and colour • Resins have greater flexibility • Better sorption capacity 	<ul style="list-style-type: none"> • Sludge disposal issues • Costly process • Used for small community systems
NPs	<ul style="list-style-type: none"> • Effective technique 	<ul style="list-style-type: none"> • Costly process
EC	<ul style="list-style-type: none"> • Separating organic matter is more efficient and quick • <i>pH</i> control is not necessary • Good efficiency 	<ul style="list-style-type: none"> • Inhibited during continuous operation by anode passivation and sludge formation on electrodes • High power consumption • Costly process

6.1 Adsorption

It is the practice of removing an element from one phase (liquid/gas) by depositing it at the interface between the liquid/gas and solid phases. By lowering the surface tension between the liquid phase and solid adsorbent, adsorbate is able to get adsorbed on the surface of solid adsorbent [2]. This method is generally used for the removal of F^- because of its better removal efficiency, selectivity, ease of handling, lower operating cost, ability to produce less sludge, and ability to regenerate used adsorbent [80].

Literatures revealed that some natural and synthetic materials in raw or treated form are used as adsorbents for the removal of F^- from potable water/wastewater. Some of these adsorbents are: neem stem charcoal [81], nanoporous Fe–Ce–Ni [82], tri-metal (Fe–Ca–Ce) oxide [83], hydrous hybrid Fe–Ca–Zr oxide [84], mechanochemically activated kaolinites [85], lanthanum-impregnated silica gel [86], heavily-weathered Tertiary soil (composed of quartz, feldspar, illite, and goethite) [87], thermally treated hydrotalcite [88], meso-structured zirconium phosphate [89], granular ferric hydroxide [90], amorphous alumina supported on carbon nanotubes [91], hydrous bismuth oxides [92], Tunisian clay minerals [93], cement paste [94,95], aluminum impregnated hierarchal web of carbon fiber [96], hydrous aluminum oxide embedded with iron oxide nanoparticle [97], bleaching powder [98], calcined Mg–Al– CO_3 layered double hydroxides [99], nano Fe–Al–Ce [100], lanthanum incorporated chitosan [101,102], granular ceramic [103], montmorillonite [104], calcite [105], *Moringa oleifera* seed [106,107], red mud [8,108], rice straw (KMnO₄-modified) [109], modified attapulgite [110], laterite [111], magnesia-amended activated alumina [112], polymer/alumina composite [113], Amberlite resin [114], Fe(III)–Al(III)–Cr(III) mixed oxide [115], activated clay [116], Zn/Al/Cl anionic clay [117], Zn/Al hydrotalcite anionic clay [118], manganese oxide coated alumina [119], schwertmannite [120,121], diatomite [122], marble waste powder [123], tea waste with Al/Fe oxides [124], fly ash [125], pumice [126], modified bentonite clay [127], natural zeolite [128], wastes from alum manufacturing process [129], bauxite [130], grapheme [131], iron ore [132], bone char [133], activated alumina [134], alum-impregnated activated alumina [135], aluminium oxide coated pumice [136], protonated chitosan bead [137], cellulose hydroxyapatite nanocomposite [138], orange juice residue [139], acid-treated bentonite [140], aluminum modified iron oxides [141], laterite [142], charcoal containing aluminum and iron oxides [143], banana peel and coffee husk [144], termite mound [145], bleaching earth [146], titanium rich bauxite [147], palm shell [148], Zr-Mn composite [149], ZrCr layered double hydroxides [150], and CeO₂–ZrO₂ nanocage [151].

Removal of F^- depends on several factors such as initial F^- ion concentration, adsorbent dose, reaction/contact time, pH, temperature, method of preparation of adsorbent, shaking speed, and presence of co-existing anions. From Table 4 it can be seen that adsorbents such as Lanthanum-impregnated silica gel, calcite, manganese oxide coated alumina, alum-impregnated activated alumina, termite mound, and rice straw can be used to remove F^- above 99%.

Table 4. Comparison among F^- removal by different adsorbents

Adsorbent	Reactor	Operational conditions		Maximum removal %	Ref.
Neem stem charcoal	Mechanical shaker 100 rpm	IFI C AD RT pH T	2 to 10 mg/L 1 to 5 g/L 0.5 to 3 h 2 to 10 25 to 55°C	94%	[81]
Nanoporous Fe–Ce–Ni	Shaker 150 rpm	IFI C AD	5 to 45 mg/L 0.04 to 0.4 g/L 0.033 to 0.75 h	98.7%	[82]

		RT pH T	2 to 12 30 to 50°C		
Tri-metal (Fe–Ca–Ce) oxide	Shaker 150 rpm	IFI C AD RT pH T	5 to 50 mg/L 0.02 to 0.25 g/L 0.083 to 1.33 h 2 to 10 30 to 50°C	≈ 98%	[83]
Hydrous hybrid Fe–Ca–Zr oxide	–	IFI C AD RT pH T	5 to 45 mg/L 0.04 to 0.5 g/L 0.083 to 0.83 h 2 to 12 30 to 50°C	96%	[84]
Mechanochemically activated kaolinites	Shaker 125 rpm	IFI C AD RT pH T	2 to 10 mg/L 10 to 50 g/L 0.167 to 1 h 3 to 11 30 to 50°C	≈ 88%	[85]
Lanthanum–impregnated silica gel	Shaker	IFI C AD RT pH T	10.45 to 104.5 mg/L 4 g/L 24 h 2 to 11 20°C	> 99.9%	[86]
Heavily–weathered Tertiary soil	Shaker	IFI C AD RT pH T	10 mg/L 33.33 g/L 48 h 7 to 7.1 25°C	30%	[87]
Hydrotalcite	Shaker	IFI C AD RT	5 mg/L 10 g/L 0.25 to 24 h 5 to 9	97%	[88]

		pH T	Room temperature		
Meso–structured zirconium phosphate	Shaker 200 rpm	IFI C AD RT pH T	1 to 10 mg/L 0.1 to 1 g/L 0.033 to 1 h 2 to 12 10 to 50°C	96%	[89]
Granular ferric hydroxide	Mixer 300 rpm	IFI C AD RT pH T	3 to 7 mg/L 5 g/L 0.167 to 0.5 h 6 to 8 Room temperature	88%	[90]
Amorphous alumina supported on carbon nanotubes	Shaker	IFI C AD RT pH T	1 to 50 mg/L 2 g/L 12 h 3 to 11 Room temperature	57.4%	[91]
Hydrous bismuth oxides	Shaker	IFI C AD RT pH T	10 to 35 mg/L 10 to 100 g/L 1 to 6 h 4 to 12 20 to 50°C	65%	[92]
Tunisian clay minerals	Shaker	IFI C AD RT pH T	2360 mg/L 33.33 g/L 2 h to 8 d 2 to 3 25°C	98%	[93]
Cement paste	Triangular flask 40 rpm	IFI C AD RT	100 mg/L 0.1 to 1 dry weight % of the liquid 48 h 1.7 to 12.5	92.6%	[94,95]

		pH T	22±0.5°C		
Hydrous aluminum oxide embedded with iron oxide nanoparticle	Shaker 280 rpm	IFI C AD RT pH T	20 to 60 mg/L 1 g/L 0.0017 to 4 h 5 to 9 25 to 50°C	90.92%	[97]
Bleaching powder	Rotary shaker 150 rpm	IFI C AD RT pH T	5 to 40 mg/L 10 to 100 g/L 1 h 3 to 12 30°C	90.6%	[98]
Calcined Mg–Al–CO ₃ layered double hydroxides	Stirrer	IFI C AD RT pH T	10 to 200 mg/L 1.1 g/L 5 to 6 h 5 to 10 30 to 80°C	98%	[99]
Lanthanum incorporated chitosan	Orbital shaker 150 rpm	IFI C AD RT pH T	5.34 mg/L 0.2 to 2 g/L 24 h 3 to 9 30°C	97%	[101, 102]
Granular ceramic	Mechanical shaker 120 rpm	IFI C AD RT pH T	5 to 50 mg/L 20 g/L 72 h 1 to 12 20 to 50°C	88.6%	[103]
Montmorillonite	Magnetic stirrer 700 rpm	IFI C AD RT pH	4 to 120 mg/L 8 g/L 0.167 to 4 h 2 to 10 25°C	65%	[104]

		T			
Calcite	Constant temperature water bath	IFI C AD RT pH T	2.97 to 2100 mg/L 33.33 g/L 72 h 6.38 to 10 22°C	99%	[105]
<i>Moringa oleifera</i> seed	Jar test apparatus 30 to 120 rpm	IFI C AD RT pH T	2 to 10 mg/L 2.5 g/L 0.017 to 1.25 h – Room temperature	92.5%	[106]
<i>Moringa oleifera</i> seed	Conical flask	IFI C AD RT pH T	0.5 to 2 mg/L 0.1 to 2.5 g/L 0.5 to 2.5 h 1 to 10 Room temperature	98%	[107]
Red mud	Conical flask 700 rpm	IFI C AD RT pH T	100 to 1000 mg/L 1 to 8.4 g/L 0.25 to 4 h 1 to 10 Room temperature	82%	[8]
Red mud	Conical flask 400 rpm	IFI C AD RT pH T	5 to 150 mg/L 5 g/L 0.25 to 9 h 2.5 to 7.3 25°C	92.33%	[108]
Rice straw	Shaker	IFI C AD RT pH T	20 mg/L 0.25 to 5 g/L 1 to 24 h 2 to 11 25 to 55°C	100%	[109]

Modified attapulgite	Thermostatic shaker 100 rpm	IFI C AD RT pH T	20 to 200 mg/L 4 g/L 48 h 6.57 20 to 40°C	87.5%	[110]
Laterite	Orbital shaker 150 rpm	IFI C AD RT pH T	3 to 50 mg/L 0.25 to 2 g/L 24 h 3 to 11 15 to 42°C	> 98%	[111]
Magnesia-amended activated alumina	Orbital shaker 90 rpm	IFI C AD RT pH T	5 to 30 mg/L 4 g/L 0 to 6 h 2 to 10 30°C	> 95%	[112]
Polymer/alumina composite	Shaking thermostatic machine 120 spm	IFI C AD RT pH T	2 to 10 mg/L 0.5 to 4 g/L 0.083 to 0.5 h 3 to 9 30 to 50°C	80%	[113]
Amberlite resin	Shaker	IFI C AD RT pH T	5 mg/L 1.25 to 10 g/L 0 to 1 h 1 to 10 25°C	92.7%	[114]
Fe(III)–Al(III)–Cr(III) mixed oxide	Thermostatic shaker 360 rpm	IFI C AD RT pH T	10 to 50 mg/L 2 g/L 0 to 2 h 3 to 10 10 to 45°C	≈ 90%	[115]
Activated clay	Shaker	IFI	13 mg/L	66%	[116]

	waterbath	C AD RT pH T	15 g/L 0 to 1 h 4 45°C		
Zn/Al/Cl anionic clay	Thermostatic shaker	IFI C AD RT pH T	4.75 to 57 mg/L 4 g/L 0 to 1 h 2.5 to 10.1 25 to 55°C	93.6%	[117]
Zn/Al hydrotalcite anionic clay	Thermostatic water bath/shaker	IFI C AD RT pH T	2 to 60 mg/L 0.1 to 4 g/L 0.5 to 16 h 4 to 10 30 to 70°C	85.5%	[118]
Manganese oxide coated alumina	Rotary shaker 80 to 90 rpm	IFI C AD RT pH T	5.13 to 32.09 mg/L 5 g/L 0 to 11 h 4 to 12 30°C	> 99%	[119]
Schwertmannite	Shaker waterbath 100 to 300 rpm	IFI C AD RT pH T	5 to 15 mg/L 1 to 4 g/L 0 to 3 h 2.6 to 11.8 25 to 55°C	≈ 83%	[120]
Schwertmannite	Thermostatic shaker 150 spm	IFI C AD RT pH T	10 to 90 mg/L 1 g/L 24 h 3 to 11 22.6 to 40°C	70%	[121]
Diatomite	Orbital shaker 40 to 200 rpm	IFI C	5 to 50 mg/L 10 to 100 g/L	91.3%	[122]

		AD RT pH T	0.083 to 2 h 3 to 11 25°C		
Marble waste powder	Orbital shaker 200 rpm	IFI C AD RT pH T	0 to 45 mg/L 3 to 15 g/L 0.5 to 5 h 3 to 10 30°C	98.66%	[123]
Tea waste with Al/Fe oxides	Vapour-bathing constant temperature vibrator 300 rpm	IFI C AD RT pH T	5 to 200 mg/L 0.4 to 8 g/L 0.017 to 5 h 2 to 11 25°C	87%	[124]
Pumice	Agitator 100 rpm	IFI C AD RT pH T	2 to 7 mg/L 5 to 20 g/L 0.5 to 3 h 4 to 9 25°C	85.75%	[126]
Modified bentonite clay	Rotary shaker 150 rpm	IFI C AD RT pH T	1 to 26 mg/L 1 to 10 g/L 24 h 5 to 9 30°C	≈ 65%	[127]
Wastes from alum manufacturing process	Magnetic stirrer 100 to 1000 rpm	IFI C AD RT pH T	5 to 40 mg/L 4 to 28 g/L 0.083 to 1 h 3 to 10 30°C	85%	[129]
Graphene	Thermostatic water-bath shaker	IFI C AD	5 to 25 mg/L 0.5 g/L 0.033 to 1.83 h	70.6%	[131]

		RT pH T	3.6 to 10.2 0 to 50°C		
Iron ore	Orbital shaker 50 to 250 rpm	IFI C AD RT pH T	2 to 29 mg/L 1 to 25 g/L 0.083 to 4 h 3 to 11 22°C	86%	[132]
Bone char	–	IFI C AD RT pH T	10 to 50 mg/L 2 g/L 0.5 to 24 h 6 to 8 20 to 40°C	24.4%	[133]
Activated alumina	Mechanical shaker 80 rpm	IFI C AD RT pH T	2.5 to 14 mg/L 4 to 40 g/L 1 to 10 h 4 to 10 –	69.5%	[134]
Alum-impregnated activated alumina	Horizontal shaker 50 rpm	IFI C AD RT pH T	1 to 35 mg/L 0.5 to 16 g/L 0 to 24 h 4 to 9 Room temperature	99%	[135]
Aluminium oxide coated pumice	Shaker 100 rpm	IFI C AD RT pH T	5 mg/L 1 to 20 g/L 0 to 24 h 3 to 11 20°C	97.8%	[136]
Protonated chitosan bead	Shaker 200 rpm	IFI C AD RT	11 to 19 mg/L 2 to 20 g/L 0.167 to 1 h 3 to 11	≈ 92%	[137]

		pH T	30 to 50°C		
Acid-treated bentonite	Shaker 400 rpm	IFI C AD RT pH T	0 to 20 mg/L 30 g/L 0 to 2 h 2.65 to 11.65 25°C	≈ 61.7%	[140]
Laterite	Incubator shaker 100 to 400 rpm	IFI C AD RT pH T	10 to 50 mg/L 0.2 to 1.2 g 0 to 3.25 h 2 to 11 20 to 40°C	1.95%	[142]
Charcoal containing aluminum and iron oxides	Shaker 300 rpm	IFI C AD RT pH T	2 to 50 mg/L 4 g/L 0 to 24 h 3 to 10 28°C	92%	[143]
Banana peel and coffee husk	Shaker 200 rpm	IFI C AD RT pH T	5 to 20 mg/L 9 to 24 g/L 0.5 to 24 h 2 to 10 Room temperature	≈ 86%	[144]
Termite mound	Horizontal shaker 200 rpm	IFI C AD RT pH T	3 to 155 mg/L 1 to 100 g/L 0 to 1 h 3 to 11 23.5 to 25.5°C	≈ 99%	[145]
Titanium rich bauxite	Shaker	IFI C AD RT pH	2 to 50 mg/L 1 to 4 g/L 0 to 6 h 3 to 9 27°C	≈ 90%	[147]

		T			
Palm shell	Incubator shaker 150 rpm	IFI C AD RT pH T	5 to 125 mg/L 0.2 g/L 0.083 to 3 h 3 to 11 25 to 45°C	> 95%	[148]
Zr-Mn composite	Shaker 150 rpm	IFI C AD RT pH T	10 to 45 mg/L 2 to 24 g/L 0.033 to 2.42 h 1 to 10 29°C	90%	[149]
ZrCr layered double hydroxides	Magnetic stirrer 600 rpm	IFI C AD RT pH T	0 to 100 mg/L 0 to 3 g/L 0 to 24 h 2.5 to 11 –	93%	[150]

(IFIC: Initial F^- ion concentration; AD: Adsorbent dose; RT: Reaction time; T: Temperature)

6.2 Precipitation/Coagulation

To facilitate the precipitation of F^- present in contaminated water, salts of Al and Ca are frequently used. The fundamental concept of the process is based on the removal of F^- after it has been adsorbed on the flocs. Although F^- can exist in several soluble forms (F^- , AlF_4^- , or AlF_2^{2+}), it is removed as precipitates of Al like $Al(OH)_{3-x}F_x$ which has to be filtered out [2]. At pH values between 5.5 and 7.5, F^- removal is most effective [51].

Many research works suggested that, the Nalgonda technique is a cheap and easy precipitation practice. It is initially tested on a small scale in Senegal, Tanzania, and Kenya. In India, this method was introduced by NEERI, Nagpur [2]. In this method bleaching powder, Al salt such as alum, and lime are added to the contaminated water which causes quick mixing, flocculation, and sedimentation and upon filtration water gets defluorinated [152]. Due to its cost-effectiveness, coagulation technique can be used in developing countries in comparison to the costlier RO system [153]. Also this technique is easy to operate and energy saving in nature [154]. In one study it is reported that for alum dose of 300 mg/L at contact time of 0.75 h and pH of 6 the F^- removal was 93.3% [155].

6.3 Nanofiltration

Due to high and specific membrane selectivity, NF technique is considered to be an effective membrane technology as compared to other defluorination processes like RO and ED [77,156]. It is a low pressure method and is used to eliminate larger dissolved solids in the order of nanometre [2]. This is considered as a bridge between RO and ultrafiltration (UF) [157,158]. Low molecular weight organic compounds as well as monovalent and divalent ions are removed by NF technique [159,160].

Cost factor, fouling, scaling, degradation, and removal of all ions present in water are some of the challenges that this technique needs to overcome [78]. It has less working pressure than RO [161]. NF membranes have a number of benefits over other purification techniques including less energy consumption, high flux, and high retention of organic contaminants and multivalent anion salts [2]. Also it has low operational expenses as compared to RO [162]. It was reported that the F^- removal by polyamide thin film composite NF membrane is more than 80% [157] and composite polyamide NF membrane retained 98% of F^- when used in the cross flow mode [158].

6.4 Reverse Osmosis

By applying hydraulic pressure greater than the osmotic pressure on the side of higher concentration, the solvent molecules are made to pass through the semi-permeable membrane to the side of lower concentration [78,163]. It works under higher pressure and discards more amounts of dissolved solids. Through molecular sieving and ionic exclusion, this technique eliminates both monovalent and divalent by 90–96% and 99% respectively. Properties of raw water, temperature, and pressure are certain factors affecting the efficiency of RO [2]. According to a report, when pH was varied between 5.5 and 7 the removal of F^- was between 45 to 90% [164]. Several other researchers reported that the removal of F^- ions using RO membranes was between 94 and 99% [165-168].

6.5 Dialysis & Electrodialysis

In dialysis, membranes are used to retain and segregate the solutes. Ions are electrochemically separated by ED, which uses DC voltage to move the ions through ion-exchange membranes. Water is made to pass through cathode and anode for concentrating the contaminated water to produce diluted or more purified water [2]. It was reported that the removal of F^- from potable water was between 80 to 90% [169] and from tap water was 92% [170].

6.6 Ion-exchange

By allowing the water to pass through ion exchanger (either anion or cation exchangers), unwanted ions can be removed. Greensand and zeolite are frequently used as cation exchangers while inorganic metal oxides are commonly used as anion exchangers [80]. By this method, water is softened and the fluoride ions are removed. The calcium form of strong acid cation exchange resin is used to remove F^- due to its high selectivity towards F^- . These resins are costly. These resins can be regenerated but the regeneration process produces F^- loaded wastes which needs further attention [77]. The efficiency of ion-exchange is low and the presence of anions affects the efficiency. So the use of cation/chelating type resins is recommended [169]. It was reported that the maximum F^- removal was 86% using a strong-base anion resin with polystyrene–divinylbenzene matrix [171].

6.7 Nanoparticles (NPs)

Nanotechnology, using NPs (1 to 100 nm) has become the active research area for the purification of contaminated water in recent years. Some distinct characteristics of NPs include smaller size, higher reactivity, large surface area, easier separation, and a large number of active adsorption sites. These characteristics make the NPs an excellent F^- adsorbent [77]. The CaO NPs have the ability to remove F^- up to 98% within 0.5 h of contact time [172]. With CuO NPs, F^- removal was reported to be more than 89% with removal capacity of 357 mg F^- /g CuO NPs [173]. NPs of gamma alumina prepared by sol–gel method with alcoholic aluminium chloride as precursor showed maximum F^- removal of nearly 23 mg/g [174].

6.8 Bio-nanocomposites and carbon-based adsorbents

Recent studies have established bio-nanocomposites as an effective adsorbent for F^- removal. These include plant biomass, industrial by-products, and agronomical wastes [153]. Carbon has strong attraction towards F^- anions, so carbon-based adsorbents are frequently used for F^- removal [175]. Low cost activated carbon prepared from *Pithecellobium dulce* [153] and *Choerospondias axillaries* [175] are effective in removal of F^- .

6.9 Electrocoagulation

An EC technique emits active coagulant precursors into solution by corroding galvanic anodes, which are typically made of aluminium or iron cations. At the cathode, electrolytic reactions produce hydrogen gas. Its implementation depends on the reactor design as well as on the electrode reliability [176]. Important benefits of EC include less sludge

generation, absence of chemical handling, less space requirement, simplicity of operation, and low initial investment [177,178]. Additionally, it eliminates refractory organic contaminants, harmful chemicals, phenolic compounds, naturally occurring organic matter, and other impurities from wastewater [179]. EC technique using aluminum electrodes has become an effective defluoridation method both for potable water and industrial wastewater [180,181]. The number of electrodes and the mode of electrode connection (either monopolar or bipolar) are both crucial factors in this process. For the removal of F^- , bipolar connection was better than monopolar connection [182]. When the initial pH was between 6 and 7, over 98% of F^- removal was observed [181]. Over 85% of F^- removal was observed at pH of 6 within a short time of 0.5 h [183].

6.10 Freeze Concentration

This technique is useful for treating many organic and inorganic industrial pollutants as well as for removing pollutants from water while ice crystals are formed [77,184]. As it involves low working temperatures, the scaling and corrosion problems are less likely to occur [185]. About 48% of F^- removal from tap water was obtained at -24°C by this technique [185]. Optimal removal of F^- was reported to be around 85% at temperatures between -15 to -20°C for deionized water spiked with fluoride [186].

6.11 Fluidized Bed Technology

Industrial gaseous effluents containing F^- are treated in a fluidized bed reactor containing some metallic bed materials. F^- from effluent react with metals present in the reactor as bed materials and form metallic F^- . Although the fluidized bed technology is not being used widely, but the researchers have observed that the fluidized bed technology is quite effective as the formation of FeF_2 , FeF_3 , and AlF_3 were confirmed through Inductively Coupled Plasma Mass Spectroscopy (ICP-MS) analysis report [187]. Thus it can be concluded that gaseous F^- gets converted to solid F^- using fluidized bed reactor which is disposed safely for land filling. Recent researches show that a new kind of fluidized bed reactor that is aerobic inverse fluidized bed biofilm reactor (AIFBBR) is being used to treat industrial wastewater containing glucose, FeSO_4 , aniline, phenol, and 4-chlorophenol as well as wastewater from brewery, refinery, steel, starch, and dairy industries [188]. Industrial liquid effluents containing F^- can also be treated in AIFBBR by suitable microbes.

6.12 Hybrid Techniques

A combination of two or more techniques is termed as hybrid treatment. These include EC and microelectrolysis, RO and NF, precipitation and adsorption, microfiltration (MF) and UF, precipitation and crystallization, EC and floatation, and metal organic frameworks (MOFs) [77]. It was reported that, 93.2% of F^- was removed by a hybrid EC-filtration unit [189]. An EC-MF hybrid technique removed 94.5% of F^- at pH of 7.9 [190]. It was reported that by employing MOFs, F^- can be removed from potable water with high efficiency [191]. It was also reported that, MOF based adsorbents have F^- removal capacity of 40 mg/g at 30°C [192] and 42.19 mg/g at 25°C [193]. NF-RO hybrid technique helped Tanzanian waters become free from F^- and natural organic matter [194].

F^- removal efficiencies of some of the methods are presented in Table 5.

Table 5. Comparison among different F^- removal methods

Method	Equipment	Operational conditions	Maximum removal %	Ref.
NF (thin film composite polyamide)	Stirred stainless steel feed reactor	35°C , pH = 10.01, 14 kg _t /cm ² , 750 L/h	> 98%	[158]
RO (thin film polyamide composite membrane, spiral wound)	RO unit	30°C , pH = 7, 2 bar, 15 L/h	95%	[168]

Ion-exchange (monovalent selective ion-exchange membrane)	ED unit	$pH = 6$ to 7 , 90 L/h, 24 V, 5 A	82 to 90.3%	[169]
ED (cation & anion exchange membrane)	Two compartment ED unit	25°C , 100 L/h, Cell constant = 0.5 cm^{-1}	92%	[170]
Nanoparticles (CaO)	Shaker	25°C , $pH = 2$ to 10 , 400 rpm	98%	[172]
Nanoparticles (CuO)	Shaker	22°C , $pH = 3$ to 13 , 0.083 to 2 h	$> 89\%$	[173]
EC	Electrocoagulator	26 to 28°C , $pH = 5$ to 9 , 450 rpm, 0.5 to 2 A, 3 h	98%	[181]
EC	Electrochemical cell	25°C , $pH = 6.4$ to 8.1 , 250 to 625 A/m^2 , 0 to 0.75 h	78 to 83%	[182]
EC	Electocogulation cell	$pH = 4$ to 10 , 0 to 1 h, 0 to 600 rpm, 0.13 to 1.38 mA/cm^2	$> 85\%$	[183]
Freeze desalination	Freezer	-5 to -30°C , 2 to 10 h	75 to 85%	[186]
Hybrid technique	EC bath (three chamber)	$pH = 7.8$, 625 A/m^2 , 0.75 h	93.2%	[189]
MOF	Shaker	25 to 35°C , $pH = 3$ to 11 , 150 rpm, 2.5 h	$> 99\%$	[193]

7. CONCLUSIONS & FUTURE PERSPECTIVES

F^{-} is abundantly found in the environment. Excess F^{-} is hazardous to human, animal, and plant health. Adsorption, RO, NF, ED, precipitation, ion-exchange, EC, freeze concentration, fluidized bed technology, biological treatment, nanotechnology, and hybrid techniques are some of the techniques used for defluoridation of water. Among these, the most widely used F^{-} removal technique is adsorption since it is inexpensive, simple to use, and has an expandable physical mechanism. The effectiveness of F^{-} removal depends on geographical, chemical, and fiscal conditions of a particular region. Given that each technology has its own benefits and drawbacks, it is suggested to select a method that should take into account the particular requirements and circumstances of the region and type of wastes generated.

Future research requirements include new low-cost materials, highly developed systems with minimal wastes, maximum utilization of generated wastes, and complete removal of F^{-} . There is a good scope for research to develop AIFBBR which has the potential to degrade F^{-} from liquid wastewater as there is no side product or hazardous waste generation. Microbes will consume F^{-} as their survival and growth.

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