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7. Defluoridation of Drinking Water Using Natural Adsorbent – Phyllanthus Emblica Wood

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Abstract:

The growing fluoride poisoning of the aquatic environment due to man-made and naturally occurring processes is one of the major problems facing the world today. Its presence is required at lower concentrations, but when it is ingested in quantities greater than 1.5 mg/L in drinking water that is primarily affected, it becomes hazardous to human health. Prolonged consumption of high fluoride levels in groundwater can have detrimental health impacts on a large number of people worldwide. It is essential for analysis and effective ways to eliminate too much fluoride from aquatic ecosystems. The widely used technique to remove fluoride is adsorption. Water quality was evaluated for 52 drinkable water samples collected from both tribal and non-tribal communities in and around Kodaikanal. Among the samples, five water samples had a higher fluoride level when compared to the BIS drinking water standard. The defluoridation process was done by using Phyllanthus emplica wood powder. About 75% of the fluoride in the aqueous fluoride solution (2 ppm) was eliminated by the Phyllanthus emblica Linn. wood powder at 0.1 g of coagulant dosage after a 2-hour treatment period at 6.6 pH. Potable water samples were also defluoridated by using - 0.1 g of Phyllanthus emplica wood powder to bring the fluoride level below the BIS standard limit. Wood powder was characterised using FT-IR and GC-MS analysis.

Keywords:

Drinking water, Fluoride, Adsorbent, Phyllanthus emblica Linn., GCMS, FTIR

7.1 Introduction:

Water is the source of life and a necessary condition for the existence of all living things as well as the progress of economic success. In recent decades, there has been a noticeable decline in the quality of aquatic resources. Groundwater contamination by fluoride is well recognized as a major problem on a global scale. Drinking water contains fluorine in its anionic form, which is the most electronegative element in the periodic table with only one oxidation state. Pure elemental fluorine is uncommon in nature due to its strong reactivity and propensity to combine with other cationic elements to produce stable compounds (Majewicz *et al*., 2020).

Surface water and groundwater fluoride concentrations and occurrence are dependent on a number of factors, including pH, total dissolved solids, alkalinity, hardness, and the geochemical composition of aquifers. These waste waters are typically generated by the production of silicon-based high-tech semiconductors, glass and ceramics, steel, uranium enrichment facilities, coal-fired power plants, beryllium extraction, photovoltaic solar cells, and superphosphate fertilizer industries. They are also produced in municipal waste incineration plants through HF emissions from the burning of fluorinated plastic, fluorinated textiles decomposition in waste sludge. However, fluorine-polluted waste water discharges are the cause of elevated fluoride concentrations in many countries worldwide. One of the toxins in drinking water that renders it unsafe for human consumption is fluoride (Manish *et al*., 2020).

The primary source of fluoride dispersing into the groundwater and surrounding ecosystem is the geological disintegration of rocks that contain fluoride. Fluoride is discharged and enters subterranean aquifers due to the presence of fluoride-rich rocks; this leads to the extensive dispersion of fluoride across the biological ecosystem (Mukherjee and Halder, 2018). Fluoride is primarily found in nature in combination forms such cryolite ($Na₃AIF₆$), fluorite (CaF₂), and fluorapatite (Ca₅ (PO₄)₃F). These substances dissolve under various chemical circumstances when they come into prolonged contact with groundwater (He *et al*., 2020). These waste waters are typically generated by the production of silicon-based high-tech semiconductors, glass and ceramics, steel, uranium enrichment facilities, coalfired power plants, beryllium extraction, photovoltaic solar cells, and superphosphate fertilizer industries.

They are also produced in municipal waste incineration plants through HF emissions from the burning of fluorinated plastic, fluorinated textiles, or CaF2 decomposition in waste sludge (Manish *et al*., 2020). The majority of residential and agricultural needs for the residents of hill stations are met by open water resources and subsurface water.

The increasing fluoride content makes the water unsafe to drink and negatively impacts human health by producing skeletal and dental fluorosis. Phytocoagulants are naturally occurring plant-based materials that can be utilized to reduce turbidity and dissolved solids during the flocculation-coagulation process of treating water and wastewater.

The element fluorine holds a special place in people's daily lives because, when present in the right amounts, it has beneficial effects like strengthening and growing bone in the teeth. Because fluoride's low concentration activates osteoblasts, products reinforced with fluoride particles such as, toothpaste to prevent dental decay are accessible on the market to benefit human society.

Furthermore, it is dangerous for a higher concentration of it to exist in any state of matter that humans can access. Frequent consumption of drinking water containing higher levels of fluoride causes skeletal and dental disorders in human bodies (Rajalakshmi *et al*., 2021).

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According to epidemiological research, drinking water is the main source of fluoride that people consume on a daily basis. Constantly using drinking water with elevated fluoride concentrations (>1.5 mg/L) can cause fluorosis in the teeth and skeleton as well as birth, reproductive, and immunological abnormalities. In addition to drinking water, fluoride can also enter the body through food, cosmetics, medications, industrial exposure, and other means (Manish *et al*., 2020).

Fluoride's strong electronegative potential strongly links it to the positively charged calcium ions in our skeletal system. Additionally, prolonged exposure to high amounts impairs our physiological metabolisms, which include the creation of DNA, proteins, minerals, and carbohydrates.

One critical quality issue is the presence of arsenic and fluoride in drinking water. Natural rock decomposition, dissolution, and dissociation, which includes fluoride-bearing minerals like topaz, apatite, amphiboles, cryolite, and fluorite, as well as industrial activities, all significantly contribute to the recruitment of fluoride in the ecosystem (Shahid *et al.,* 2020).

The effect *of Phyllanthus emblica* L. wood powder elimination of heavy metals, fluoride, turbidity, hardness, and chemical oxygen demand (COD) in contaminated water and found that the main reason for the wood powder's ability to clarify was because it contained Minerals (sodium, magnesium, potassium, zinc, iron, phosphorus, and calcium) make up 46% of the dry fruits. Carbohydrates (sodium, magnesium, potassium, fibers, and glycosides) make up 70 75% of the dry fruits. Proteins (2.5 3.5%), which contain a variety of amino acids (alanine, lysine, proline, glutamic acid, cysteine, and aspartic acid), lipids (1.5 2.0%), and 2.5 3.5% comprise a wide range of other substances.

Because fluoride has both positive and negative effects on human health, its presence in drinking water is a temporary disaster. Furthermore, because natural plant-based products are readily available around-the-clock, they are inexpensive (Ahmad *et al*., 2021).

The most effective substance for removal is the magnetic bioadsorbent. High efficacy magnetic particles are used to effectively remove harmful heavy metals from water and other aquatic settings.

The adsorption phenomenon is a very effective and practical method that is frequently used to clean up pollutants, such as hazardous metals (Pb2+, Cd2+) found in aquatic environments. Indian gooseberry Bark (*Phyllanthus emblica*) is a good source of adsorbents, which can be used in place of water contaminants can be effectively removed from a pollution by using composite magnetic particles as an adsorbent (Deshmukh *et al*. (2021).

The study was intended to defluoridate potable water using *Phyllanthus emblica* Linn. wood because both tribal and non-tribal residents of Kodaikanal villages rely on the accessible freshwater zones from the hills for a variety of domestic and agricultural purposes.

However, the currently accessible hill water sources may contain several salts and mineral components emerged due to anthropogenic and natural environmental accomplishments.

7.2 Materials and Methods:

7.2.1 Drinkable Water Sample Collection:

Table S1 details the 52 sampling sites in and around Kodaikanal that were gathered from both tribal and non-tribal areas. During the summer season, starting in May 2017, fresh drinking water samples were collected in pre-cleaned and sanitized bottles for this investigation.

The water samples were safely transferred to the laboratory and appropriately labeled; to prevent physico-chemical changes, all of the water samples were kept at 4 degrees Celsius and used in subsequent experiments.

7.2.2 Analysis of Fluoride in Drinking Water Samples:

All drinking water samples were subjected to fluoride analysis as per standard method APHA, (2012) in order to find the fluoride contaminated samples. Each chemical used in this research was supplied by Merck and Sigma Aldrich. Deionized and double-distilled water were utilized for the entire experiment.

The BIS drinking water fluoride standard was used to compare all the water samples.

7.2.3 *Phyllanthus Emblica* **Linn. Wood Powder Preparation for Fluoride Removal:**

The wood, *Phyllanthus emblica* Linn., was collected from the Combai (tribal area) in Kodaikanal hill, Tamil Nadu, India. Mature and healthy wood were extensively rinsed with distilled water after being thoroughly cleansed with running tap water to remove any dirt particles that had become adhered to the wood's surface. The wood was processed with rice mill equipment and allowed to air dry in the shade. The wood particle powder has a smooth, coarse, and fine texture, and the wood powder was sieved (0.075 mm). The dried powder was then kept in an airtight container for use in drinking water defluoridation testing.

7.2.4. Research on the Use of Phyllanthus Emblica Linn. Wood To Remove Fluoride Ions from Aqueous Solutions:

Sodium fluoride salt was dissolved in deionized water to create an aqueous fluoride stock solution. 100 mL of working fluoride solutions at various concentrations, including 1, 2, 3, 4, and 5 ppm, were created using the stock solution. Each conical flask contained 0.05, 0.1, 0.15, 0.2, 0.25, and 0.3g of wood powder in order to test the removal of fluoride ions from aqueous solutions. After thoroughly mixing the added wood powder, the mixture was left alone for two hours. After passing through Whatmann No. 1 filter paper, the aqueous solution was tested using the fluoride removal assay. There was a control conical flask kept for every concentration of aqueous fluoride. The experiment was repeated multiple times to find the optimal dose of wood coagulant for each aqueous fluoride concentration, and the percentage of fluoride removal was recorded from each concentration.

7.2.5 Studies on Fluoride Removal from The Potable Water Samples Using Phyllanthus Emblica Linn. Wood:

A 100 ml of each fluoride contaminated drinking water samples (Keelpoomi, Naidupuram, Villpatti, Pallangi and Perumal malai) were treated with 0.1 g of *Phyllanthus emblica* Linn. wood, which was screened and confirmed from aqueous fluoride removal studies. The wood powder was added into conical flasks filled with each water samples, mixed thoroughly and left undisturbed for 2 h. The treated water samples were filtered using Whatmann No.1 filter paper before being tested for fluoride removal. All the treatment experiments were repeated thrice to confirm the percentage of fluoride removal.

7.2.6 Characterization of *Phyllanthus Emblica* **Linn. Wood**

A. GC–MS analysis of the *Phyllanthus emblica* **Linn. Wood:**

The phytocompounds (1 μl) extracted from *Phyllanthus emblica* Linn. wood powder with methanol were employed for GC–MS analysis. Clarus 500 GC The experiment was performed using a Perkin Elmer system that included an AOC-20i auto sampler and a gas chromatograph connected to a mass spectrometer (GC/MS). The mass spectrum created in

Figure 7.1 shows analyse using the data using National Institute of Standards and Technology (NIST) database, which consists many hundreds of patterns. The name, molecular weight, and structure of plant wood components were determined using the NIST Ver. 2.1 MS data library.

B. FTIR characterization of *Phyllanthus emblica* **Linn. wood material before and after the studies on the treatment of drinking water:**

Phyllanthus emblica Linn. wood active functional groups matter participated in the fluoride removal from drinking water samples were identified through FTIR spectral analysis (before and after treatment). The characterization of the dried wood powder used for treatment studies was done by using FTIR instrument (Model: Spectrum Perkin RXT) by directly placing the Potassium Bromide crystals over the sample. The mass spectrum obtained from FTIR in the mid IR region of 450–4000 cm−1 was documented using ATR (Attenuated Reflectance Technique) in transmittance mode of analysis.

7.3 Results and Discussion:

7.3.1 Fluoride Analysis:

The examined samples had fluoride values ranging from 0.1 ± 0.15 to 1.99 ± 0.22 mg/L. Five samples had higher than recommended levels of fluoride such as Naidupuram S5 (1.81 mg/L), Pallangi S7 (1.99 mg/L), Villpatti S9 (1.89 mg/L), Perumalmalai S17 (1.97 mg/L), and Keelpoomi S4 (1.86 mg/L). The highest allowable fluoride concentration, as per the BIS standard, is 1.1–1.5 mg/L. Furthermore, Memon *et al*. (2021) studied the fluoride concentrations in Iran's Shusha Aquifer, which ranged from 0.12 to 2.17 mg/L. Soil and rock is the source of fluoride found in drinking water found in pools, rivers, and streams. The primary cause of the elevated fluoride levels in these water samples is the mineral's leaching, weathering, and erosion from the hills' granite, thus, the higher level of fluoride content that was acquired from the drinking water samples. Therefore, eliminating fluoride and keeping it under guidelines may be good for human health. Various methods of removing fluoride have been proposed and implemented worldwide. The Nalgonda procedure is said to be the water defluoridation method that is most frequently used. NEERI (National Environmental Engineering Research Institute, Nagpur, India) produced this. Using this procedure, the water is mixed with several chemical agents, such as lime, bleaching powder, and aluminum sulfate, and left for three hours to allow the defluoridation process to occur. Despite being a widely used approach, there have been significant negative reports as well (Rajalakshmi *et al*., 2021).

Levels of this metals, in drinking water that are too much or too low are dangerous. A comparable finding was reported by Abreu *et al*., (2019), which resulted in a surfeit of fluoride (7 mg/L) in cooking water. One of the main explanations for the elevated fluoride levels in samples is due to leakage, weather, and wearing a way of fluorine from the hills' big stone. As a result, removing fluoride and limiting it to safe levels could be beneficial to well-being of human health. Globally, there are many reduction techniques of fluoride. In India, fluorosis is a serious threat, due to the intake of fluoride polluted water which accounts for the suffer of 62 million people, including 6 million children.

7.3.2 Effect of *Phyllanthus Emblica* **Linn. Wood Adsorbent on Removal of Fluoride from Aqueous Solutions:**

Phyllanthus emblica Linn. wood adsorbent was used to remove fluoride ions at different concentrations of the aqueous fluoride solutions. A minimum of 17% to a maximum of 67% of fluoride ion was reduced by different doses of wood adsorbent used in this study. When the wood adsorbent dose was increased from the original dosage, the removal of fluoride ion from aqueous fluoride solutions was increased. After a particular amount of adsorbent addition, the fluoride removal percentage from the aqueous solution was stable. Further, the addition of wood adsorbent lowered the percentage of fluoride ion removal and was poor.

Among these adsorbent doses 0.05, 0.1, 0.15, 0.2, 0.25 and 0.3 g added, 0.2 g of wood adsorbent dose has removed the highest percentage (40, 47, 55, 65 and 67%) of fluoride ion from all the above prepared synthetic fluoride solutions. This optimum dose (0.2 g) removed the maximum percentage of fluoride ion (67%) from 2 ppm solution than the other concentrations of aqueous solutions studied. Similar outcomes were noticed by Rajalakshmi *et al*., (2021) when they used *Strychnos potatorum* seed powder to remove fluoride.

The maximum clearance percentage (64%) was observed at 0.2g of the powder. The effectiveness of zeolite NaA in the sorption of fluoride from aqueous solution was demonstrated by Mukherjee *et al*. (2018). When the fluoride content was 10 mg/L, the pH of the solution was 5, the temperature was 45 °C, and the adsorbent dosage was 105 mg, the maximum removal of 90% was attained. The contact period was 4 hours. *Phyllanthus emblica* Linn. activated carbon has been examined by Veeraputhiran and Alagumuthu (2011) for the removal of fluoride from water. At neutral pH and 75 minutes of contact time, the highest fluoride removal capacity of 82.1 percent was achieved with an adsorbent

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dosage of 0.75 g and a 3-ppm starting fluoride concentration. Deshmukh *et al*., (2021) have stated that the *Phyllanthus emblica* (Amla) bark contains amino acids, carbohydrates, furosin, gallic acid, and corilagain, among other phytochemicals, Gallic acid's polyphenolic groups are what cause various metal ions to be reduced. Akafu *et al*., (2019) fluoride exposure over the allowed quantity for a longer period of time causes both dental and skeletal fluorosis. Their research also included the removal of fluoride from drinking water by sorption using diatomite modified with aluminium hydroxide, and they discovered that under ideal conditions, the maximum fluoride removal was 89.4%, indicating that aluminium hydroxide-modified diatomite can be used as an efficient, inexpensive, and environmentally friendly adsorbent. Jenifer et al. (2016) discovered that the element fluoride ion in Kanyakumari district bore well water was below the authorised BIS levels in all water samples, ranging from 1-1.5 mg/L.

7.3.3 Effect of *Phyllanthus Emblica* **Linn. Wood Adsorbent on Removal of Fluoride from Drinking Water:**

Potable water samples from Naidupuram, Villpatti, Pallangi, Perumal malai, and Keelpoomi were found to be contaminated with fluoride. The fluoride concentration was higher than the BIS standard. The contaminated samples were defluorinated by 0.2 g of *Phyllanthus emblica* Linn. wood powder.

The wood adsorbent effectively reduced 58% (Naidupuram), 52% (Villpatti), 52% (Pallangi), 61% (Perumal malai) and 44% (Keelpoomi) of fluoride from the polluted water samples. The highest fluoride removal (61%) was appeared in Perumal malai water sample.

Using powdered *Strychnos potatorum* seed, Rajalakshmi *et al*. (2021) reported removing 75% of fluoride from drinking water. Good coagulant protein can be found in *Strychnos potatorum* L. seeds.

Through surface charge neutralization, flocculation, and coagulation, the polyelectrolyte and cationic amino acids of the protein (15.1 kDa) may have drawn the negatively charged fluoride ions. It's possible that this process eliminated the fluoride ions from the tainted water samples in the end.

The reason for the drop in the fluoride removal percentage after two hours of treatment could be that the active sites of the seed coagulant had reached equilibrium. Arunkumar *et al*. (2019) published a study on the removal of contaminants from water samples using *Strychnos potatorum* L. seed protein (12 kDa).

Suneetha and Ravindranath (2019) state that the best powders for ammonia adsorption are those derived from the leaves and bark of *Phyllanthus niruri, Annona squamosa, Calotropis gigantean, Tridax procumbens, Morinda tinctoria,* and *Azadiracht indica.*

These powders also yield the highest percentages of ammonia removal, but they are pH sensitive and dependent on the sorption concentration and equilibrium time. With this biosorbent dosage, ammonia may be eliminated over 90% at pH $\frac{1}{4}$ 5, optimal equilibrium time, and sorbent concentrations.

Panchu *et al*., (2022) investigated the use of *Phyllanthus Emblica*, *Strychnos Potatorum*, and *Moringa Pterygosperma* as adsorbents in a batch adsorption procedure to remove fluoride and Methlene Blue dye from water. At 30 minutes (pH 9), these adsorbents corresponding maximal fluoride adsorption capacities were 23 mg/g, 24 mg/g, and 31 mg/g.

An experiment was carried out by Mengistie *et al*. ([2006) to determine the effects of a modified handmade filter on temperature, pH, turbidity, fecal coliform, and fluoride.

The study's conclusions imply that significant amounts of physical, chemical, and biological contaminants might be eliminated by filtering fluoridated raw water with the material used in the investigation. The chelating capability of *Phyllanthus emblica* Linn. wood, according to Sathis *et al*., (2012), may be responsible for the lowering of magnesium levels in water.

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Magnesium salts are more soluble than calcium, raising the hardness of water and imparting an unpleasant taste. If consumed in large amounts, they may cause laxative effects. Side effects include renal failure, respiratory depression, and cardiac arrest.

As a result, the wood of *Phyllanthus emblica* Linn. has a positive impact on magnesium levels in this study. According to Thamaraiselvi et al., (2023), tamarind seeds can remove 60 percent of fluoride from drinking water.

Table.7.2. Effective removal of Fluoride from potable water samples using *Phyllanthus emblica* **Linn.**

7.3.4 Characterization of *Phyllanthus emblica* **Linn. Wood Material:**

A. GC-MS analysis of *Phyllanthus emblica* **Linn. wood extract:**

GC-MS examination of *Phyllanthus emblica* Linn. methanol extracts revealed the presence of 25 phytocompounds, respectively. The obtained phytochemical compounds in the wood methanol extracts of *Phyllanthus emblica* L.

Twenty-five active compounds were identified from wood methanol extracts of *Phyllanthus emblica* L.

The active compounds were 3-O-Benzyl-d-glucose, alpha D-Glucopyranoside, 4H-Pyran-4-one Cyclohexanamine, Furancarboxaldehyde, cetyl beta d-mannose, Glucopyranoside, Azetidin, aminohexylurea, Imidazole, Phthalic acid, Pyridine-2,4-diol, Octacosanoic acid, Ascorbic acid 2,6-dihexadecanoate, Cyclopropaneoctanoic acid, 4H-Pyran-4-one, Methyl 3-acetamido-4:6-phenylisopropylidene -3-deoxy-alpha-d-altropyranoside, alpha-D-Glucopyranoside, alpha-d-altropyranoside, 1,2,3-Benzenetriol Pyrogallol, thiophosphatoethyl aminohexylurea, Alpha-D-Glucopyranoside, Estra-1,3,5(10)-trien-17betanol, 1-Heptadec-1-ynyl-cyclopentanol, Oxacycloheptadec-8-en-2-one, 9- Octadecenal, Ethyl 1-thio-alpha. -l-arabinofuranoside and Octadecanoic acid were the major components in the wood extracts. Quranayati, *et al.* (2022) used methanol extracts of Phyllanthus bark to produce similar gcms results.

According to Asmilia *et al*., (2020), the ethanolic extract of *Phyllanthus emblica* Linn. leaves included 22 chemical components, with decanoic acid being the most common (22.93 %). These chemical elements are important for the efficacy of traditional medications, especially as antioxidants and antimalarials.

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Figure 7.5: GC-MS chromatogram of methanol wood extract of *Phyllanthus emblica* **L.**

B. FTIR Analysis of *Phyllanthus Emblica* **Linn. Wood Material After the Treatment of Drinking Water**

The FTIR analysis of *Phyllanthus emblica* Linn wood after fluoride treatment shows the band from 629 cm⁻¹ to 3455 cm-¹. 629 cm⁻¹ band represent = C-H bending of Aliphatic Hydrocarbon, 1120 cm⁻¹ represent SO₂ symmetric stretching of Sulfur Compound, 1416 cm⁻¹ represent Si-C6H5 stretching of Silicon Compound, 1641 cm⁻¹ band shows Pyridine C=N stretching, C=C stretching of nitrogen containing compound and 3455 cm-¹ band shows Si–C6H5 stretching of Silicon Compound.

After treatment, the spectrum shows the absence of Second overtone N–H stretching and second overtone O–H stretching of Organic Compound compared to untreated *Phyllanthus emblica* linn. wood powder which might have been involved in the removal process.

Table 7.4: FTIR analysis of *Phyllanthus emblica* **Linn after fluoride treatment**

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Figure 7.6: FTIR analysis of *Phyllanthus emblica* **Linn after fluoride treatment**

7.4 Conclusion:

The *Phyllanthus emblica* Linn. wood successfully eliminated fluoride from drinking water samples and aqueous solutions. A FTIR study of the wood revealed the presence of phenol chemicals, alcohol, amine, and sulfur molecules. Through the adsorption mechanism, these functional groups actively contributed in the water defluoridation process.

Thus, in 120 minutes and two hours, the wood powder effectively eliminated 67% of the fluoride from drinkable water*.* GCMS studies also revealed 25 phytochemcals. The defluoridation property may be favored by -d-mannose, glucopyranoside, azetidin, aminohexylurea, imidazole, phthalic acid, and pyridine-2,4-diol. Hence, this study concludes that *Phyllanthus emblica* Linn. wood powder could be utilized to efficiently defluoridate drinkable water by substituting chemical agents with their adsorption mechanism and associated treatment techniques to clean drinking water.

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