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Use of Maize husk fly ash as an adsorbent for removal of fluoride

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Abstract -.Fluoride related health hazards are a major environmental problem in many regions of the world. Literature review reveals that, India is among the 25 nations around the globe, where health problem occurs due to the consumption of fluoride-contaminated water. In India, 17 states have been identified as epidemic for fluorosis and Orissa is one of them. The fluoride contamination in Orissa is wide spread, where 10 district out of 30 have excess of fluoride in ground water. More than 60% of our fluoride demand is fulfilled by the consumption of drinking water. Excess of fluoride (>1.5 mg/L) in drinking water is harmful to the human health. The physiological effects of fluoride upon human health have been studied since the early part of 20th century. Therefore in view of finding out a method to remove the fluorine from water, extensive research has been carried out by many people

***Key Words:** , Fluoride , Maize husk fly ash, adsorbent*

1Introduction.

Fluorine, a fairly common element of the earth's crust, is present in the form of fluorides in a number of minerals and in many rocks. Excess fluoride in drinking-water causes harmful effects such as dental fluorosis and skeletal fluorosis. The permissible limit of fluoride level is generally 1 mg/l. The high fluoride levels in drinking-water and its impact on human health in many parts of India have increased the importance of defluoridation studies. The fluoride-bearing minerals or fluoride-rich minerals in the rocks and soils are the cause of high fluoride content in the groundwater, which is the main source of drinking-water in India. Fluorine is the chemical element with atomic number 9, represented by the symbol 'F'. It is the lightest element of the halogen column of the periodic table and has a single stable isotope, fluorine-19. At standard pressure and temperature, fluorine is a pale yellow gas composed of diatomic molecules, F2. Fluorine is rare compared to other light elements. In Earth's crust, fluorine is more common, being the 13th most abundant element. Adsorption is an efficient and economically viable technology for the removal of fluoride. Recently, many naturally occurring materials such as activated carbon from plant materials, egg shell,bone-char, Tamarind seed, rice husk, limestone and some commercially available adsorbent such as Activated Alumina, calcium hydroxide [$Ca(OH)_2$], calcium chloride [$CaCl_2$] , and calcium sulphate [$CaSO_4$] have been used for removal of fluoride. However, the alternative sorbants have not displayed significant fluoride removal capacities and, thus, alumina still remains a valuable material to study and pursue. Despite decades of application-based research, the underlying science and specific mechanisms behind fluoride sorption to alumina based sorbants is still unclear Fluorides are one of three important hazardous chemical in addition to arsenic & nitrate. Fluoride is highly reactive and is found naturally as CaF_2, It is an essential constituent in minerals like topaz, tluorite, lluorapatite, cryolite, phosphorite, theorapatite, etc. The fluoride is found in the atmosphere, soil and water. It enters the soil through weathering of rocks, precipitation or waste runoff. Surface waters generally do not contain more than 0.3mg/l of fluoride unless they are polluted from external sources. Though drinking water is the major contributor (75- 90% of daily intake), other sources of fluoride poisoning are food, industrial exposure, drugs, cosmetics, etc. fluoride which causes large- scale health problem by exposure through drinking water. Fluoride in drinking water may be beneficial or detrimental depending on its concentration & total amount ingested .Fluoride is beneficial especially to young children below 8 yrs of age when present within permissible limits of 1.0-1.5 mg/l for calcification of dental enamel. Indian standards for drinking water recommended an acceptable fluoride concentration of 1.0 mg/l & an allowable fluoride concentration of 1.5 mg/l in potable water (CPHEEO, 1984)

2 PROBLEM STATEMENT:

A number of studies have reported on the acute effects of fluoride exposure following fluoridation overdosing. However, the effects of long-term exposure to naturally occurring fluoride from drinking-water and other environmental sources are the major concern with regard to human health. A large number of epidemiological studies have been conducted in many countries concerning the effects of long-term exposure to fluoride. Information from countries of dental or skeletal fluorosis has been documented.

Therefore, there is a need of a thorough study on influence of fluorine on nature and to find out suitable methods of Defluoridation.

Fly ash is the major solid waste by-product. It is produced as a fine residue carried off in the flue gases with relatively uniform particle size distribution in the 1 to 100 μm range. The main components of fly ash are silica, alumina, iron oxides, calcium oxide, and residual carbon. The fineness of the fly ash particles and the inherent large surface area (1 to 6 $m^2\ g^{-1}$), together with the content of unburnt carbon, make it a good candidate for utilization as an inexpensive sorbent.

A literature survey revealed that fly ash has been used for removing heavy metals and radio nuclides from aqueous solutions, for treatment of wastewaters to remove organic compounds and color, as a coal desulphurization agent, and together with hydrated lime for SO_2 removal from flue gases.

2.1 OBJECTIVES:

The main objectives of this study are:

1. To evaluate the efficiency of fly ash from thermal power plant and maize husk fly ash for removal of fluoride from water.
2. Optimization of the different parameters to be varied, to find the equilibrium values, in order to get maximum efficiency.
3. Validation of the results through modeling using adsorption isotherms.

2.2 SCOPE OF THE STUDY

Both quality and quantity of water supply plays a significant role for the protection of public health. The need for the improvement of surface water supply on which the majority of the rural population in developing countries still depends is well recognized. Fluoride is one of the very few chemicals that have been shown to cause significant effects in people through drinking-water. Fluoride has beneficial effects on teeth at low concentrations in drinking-water, but excessive exposure to fluoride in drinking-water, or in combination with exposure to fluoride from other sources, can give rise to a number of adverse effects. It is a challenging task though a feasible solution to introduce the traditional purification methods such as the use of low cost adsorbents. Researchers have found out many natural adsorbents to treat water and many of them are used all over the world. In the present study the fly ash obtained from thermal power plant and the fly ashes obtained from Maize husk are used as adsorbents which are not popular in India even though they are in abundant quantity all over the country.

The study was undertaken along the following lines.

1. Performance evaluation of Thermal power plant fly ash (TPP fly ash) and Maize husk fly ash(MH fly ash) in Batch study, in terms of fluoride , by carrying out the experimental work for five different parameters, viz. Contact time, p^H, Adsorbent dose, Stirring rate and Initial fluoride concentration.
2. Optimization of both the adsorbents for the five parameters varied, to obtain maximum efficiency of fluoride removal from water. The scope of this phase of the study is to reduce the fluoride level, so that the ill effects due to fluoride concentration can be reduced.
3. Infra red Spectra study to probe into the nature of the adsorbing action of both the fly ash used.
4. Validation of the results obtained, in terms of fluoride removal efficiency (percentage), using adsorption isotherms, viz. Langmuir, Freundlich, Temkin and Redlich- Perterson isotherm models.

3 Effects on humans

A number of studies have reported on the acute effects of fluoride exposure following fluoridation overdosing. However, the effects of long-term exposure to naturally occurring fluoride from drinking-water and other environmental sources are the major concern with regard to human health. A large number of epidemiological studies have been conducted in many countries concerning the effects of long-term exposure to fluoride. Information from countries of dental or skeletal fluorosis has been documented.

3.2 Effects on teeth

The beneficial and the detrimental effects of fluoride naturally present in water were well established by the early 1940s. High levels of fluoride present in concentrations up to 10 mg/l were associated with dental fluorosis (yellowish or brownish striations or mottling of the enamel) while low levels of fluoride, less than 0.1 mg/l, were associated with high levels of dental decay, although poor nutritional status is also an important contributory factor. Concentrations in drinking-water of about 1 mg/l are associated with a lower incidence of dental caries, particularly in children, whereas excess intake of fluoride can result in dental fluorosis. In severe cases this can result in erosion of enamel. The margin between the beneficial effects of fluoride and the occurrence of dental fluorosis is small and public health programmes seek to retain a suitable balance between the two. The level of dental caries (measured as the mean number of Decayed, Missing or Filled teeth) falls from seven at a fluoride concentration of 0.1 mg/l to around 3.5 at a fluoride concentration of 1.0 mg/l. As fluoride concentration increased further (up to 2.6 mg/l) dental decay continues to fall, but only slightly. Conversely, dental fluorosis increases as fluoride concentration increases. At a fluoride concentration of 1 mg/l about 20 per cent of children have evidence of dental fluorosis but this fluorosis is of a mild degree of severity and would not be cosmetically obvious to the children or their parents. Thus the evidence suggested that, at least for fluoride naturally present in water, the optimal level of fluoride for a temperate climate was around 1 mg/l; this concentration was associated with a substantial resistance to tooth decay but with only a small and cosmetically insignificant increase in the prevalence of dental fluorosis.

Dental fluorosis is a cosmetic effect that ranges in appearance from scarcely discernible to a marked staining or pitting of the teeth in severe forms. It is caused by an elevated fluoride level in, or adjacent to, the developing enamel. Thus, it follows that dental fluorosis can develop in children but not adults. Dental fluorosis in an adult is a result of high fluoride exposure when the adult was a child or adolescent There are a variety of ways of describing dental fluorosis.

Thus, for example, in China some 38 million people are reported to suffer from dental fluorosis and 1.7 million from the more severe skeletal fluorosis. In India, around one million people suffer from serious and in capacitating skeletal fluorosis. Using the Chinese dental:skeletal fluorosis ratio, India could therefore have up to 20 million dental fluorosis sufferers, and in fact, fluorosis affects an estimated 25 million people in India. Thus in India and China alone over 60 million people may be affected and, when other populations in Africa and the eastern Mediterranean in particular are taken into account, the global total may exceed 70 million

3.3 Skeletal effects

Endemic skeletal fluorosis is well documented and is known to occur with a range of severity in several parts of the world, including India, China and northern, eastern, central and southern Africa. It is primarily associated with the consumption of drinking-water containing elevated levels of fluoride but exposure to additional sources of fluoride such as high fluoride coal is also potentially very important. This is compounded by a number of factors which include climate, related to water consumption, nutritional status and diet, including additional sources of fluoride and exposure to other substances that modify the absorption of fluoride into the body. Crippling skeletal fluorosis, which is associated with the higher levels of exposure, can result from osteosclerosis, ligamentous and tendinous calcification and extreme bone deformity. Evidence from occupational exposure also indicates that exposure to elevated concentrations of fluoride in the air may also be a cause of skeletal fluorosis.

Although there are a large number of epidemiological studies available, the data are such that it is difficult to determine a clear exposure–response relationship. One possible feature of fluorosis is bone fracture, although some studies have reported a protective effect of fluoride on fracture. In an epidemiological study in China the relationship between fluoride intake via drinking-water and all other sources, and all fractures, followed a U shaped dose response with higher rates of fracture at very low intakes below 0.34 mg/l and high intakes above 4.32 mg/l (total intake 14 mg per day). It was concluded that for a total intake of 14 mg per day there is a clear excess risk of skeletal adverse effects and there is suggestive evidence of an increased risk of effects on the skeleton at total fluoride intakes above about 6 mg per day

3..4 Cancer

Studies of occupationally exposed populations, primarily from aluminium smelting, have reported an increased incidence of, and mortality from, lung and bladder cancer and from cancers in other sites. However, the data are inconsistent and in a number of studies the results can be more readily attributed to exposure to other substances than fluoride. There have also been a significant number of epidemiological studies examining the possible association between various cancers and exposure to fluoride in drinking-water. However, in spite of the large number of studies conducted in a number of countries, there is no consistent evidence to demonstrate any association between the consumption of controlled fluoridated drinking-water and either morbidity or mortality from cancer.

3.5. Other possible health effects

A number of epidemiological studies have been carried out to examine other possible adverse outcomes as a consequence of exposure to fluoride, either from drinking-water or as a consequence of occupation. Studies on the association between exposure of mothers to fluoride in drinking-water and adverse pregnancy outcome have shown no increased risk of either spontaneous abortion or congenital malformations. No reasonable evidence of effects on the respiratory, haematopoietic, hepatic or renal systems have emerged from studies of occupationally exposed populations that could be attributed specifically to fluoride exposure. In addition, such studies have failed to produce convincing evidence of genotoxic effects. The majority of fluoride is excreted via the kidneys. Thus it is reasonable that those with impaired renal function might be at greater risk of fluoride toxicity than those without. In discussing this point it was concluded that the data were too limited to permit any quantitative evaluation of possible increased sensitivity due to impaired kidney function

3..6 Acute effects

A number of overdosing incidents have occurred, mostly in small water supplies, which practice artificial fluoridation. With well designed fail-safe equipment and working practices overdosing incidents can be avoided. Where incidents of acute intoxication have been reported following overdosing in water supplies, fluoride levels have ranged from 30–1,000 mg/l. To produce signs of acute fluoride intoxication, it is estimated that minimum oral doses of at least 1 mg fluoride per kg of body weight are required. Indeed, such doses could be expected from water with a fluoride content of approximately 30 mg/l.

3..7 Psychological Fluorosis

Fluorosis may above all affect people socially and psychologically. Many fluorotic persons tend to hide their teeth and to constrain their smiles, and that may affect their personalities, their behaviour and their social potential for life time.

This phenomenon is very well known in some regions and may be called "**Psychological Fluorosis**". This type of fluorosis is still to be studied and quantified professionally.

4.1 Materials Collection & Sampling

Materials are the main constituents which are required to perform experimentation. The various materials used in this study are; water, natural adsorbents (Thermal Power Plant fly ash, Maize husk fly ash), zirconyl acid reagent, spands solution and reference solution. The descriptions of these materials are as below.

4.1.1 Fly ash:

Fly ash is the major solid waste by-product from coal-fired power plants. It is produced as a fine residue carried off in the flue gases with relatively uniform particle size distribution in the 1 to 100 μm range. The main components of fly ash are silica, alumina, iron oxides, calcium oxide, and residual carbon. The fineness of the fly ash particles and the inherent large surface area (1 to 6 m^2 g^{-1}), together with the content of unburnt carbon, make it a good candidate for utilization as an inexpensive sorbent.

A literature survey revealed that fly ash has been used for removing heavy metals and radionuclides from aqueous solutions, for treatment of wastewaters to remove organic compounds and color, as a coal desulphurization agent, and together with hydrated lime for SO_2 removal from flue gases.

There is also evidence that the capital and operating costs of wastewater treatment by fly ash to remove turbidity, fluoride, and to reduce COD, are lower than those by conventional lime precipitation.

In view of the advantages of fly ash as a low-cost sorbent, it seemed worth-while to study its efficiency in removing fluoride ion from high-fluoride waters.

Two Different Types of flyash were used for the present study. One of the flyash named as Thermal power plant flyash (TPP flyash) was collected directly at the electrostatic precipitator of the Dirk India Pvt. Ltd., Nashik, Thermal Power Plant and the other as a residual Maize Husk flyash (MH flyash). The grain size of both the ash ranged between 1-90 µm with a mean diameter of 20-30 µm.

Method of Preparation of fly ash from Maize Husk is detailed as below:

The method used to produce the corn ash started from the completion of the growing season, corn stalks were hand-harvested by cutting the stalk approximately 15 cm (6 inches) above ground level and then cleaned to remove soil particles. The corn biomass, termed corn husk, included the corn cobs and kernels in some amount. The corn husks were then air dried and coarsely chopped before shipment to the lab. The corn ashes were produced at temperatures below 700ºC (1,292ºF) .Simple open burn procedures typically occur between 500ºC (932ºF) and 600ºC (1,112ºF) [50]. The dried corn husks were burned to 500ºC (932ºF) to obtain the fly ash. It was then passed

Table1: Chemical analysis of TPP fly ash and MH fly ash

Constituent (Chemical characteristics)	**TPP Fly ash**	**Maize Husk Fly ash**
	Amount (%)	Amount (%)
SiO_2	52.70	38.33
k_2O	---	27.58
CaO	7.20	7.83
MgO	---	5.01
P_2O_5	---	4.53
Cl	---	3.02
SO_3	---	1.72
Fe_2O_3	8.40	0.47
Al_2O_3	21.90	0.22
LOI	9.10	11.4

4.2 METHODOLOGY

Experimental work of this study has been divided into following phases. They are

• Adsorbance procedure by Batch study.

1. Effect of contact time on fluoride removal.
2. Effect of p^H on fluoride removal.
3. Effect of Adsorbent dose on fluoride removal.
4. Effect of stirring rate on fluoride removal.
5. Effect of initial fluoride concentration (IFC) on fluoride removal.

• Validation of results through Modeling (Langmuir, Freundlich, Temkin, Redlich-perterson Models) for fluoride removal

• Infra Red (IR) Spectra Study to determine the presence of adsorption friendly functional groups in two adsorbents used.

4.2.1 Batch study

Batch experiments were performed for the determination of equilibrium time, optimum p^H, optimum dosage, optimum stirring rate and initial fluoride concentration and selection of a best fitted adsorption isotherm. All the experiments were carried out in 100 ml glass jar with 50 ml test solution at room temperature (29 ± 2°C). The jar, along with known volume of test solution of fixed concentration and 1.0 g of the adsorbent at neutral p^H, was shaken in mechanical stirrer at 400 rpm to study the equilibration time for maximum adsorption of fluoride.

The batch study was performed to determine the optimum conditions and to study the effect of p^H, adsorbent dose, contact time, stirring effect and initial fluoride concentration on the test solution. The effect of p^H on fluoride was studied by adjusting the p^H of test solution using 0.1N HCl or 0.1N NaOH on fixed quantity of adsorbent, while effect of adsorbent dose and contact time was studied by varying dose and contact time, respectively. At the end of the desired contact time, the sample was filtered using Whatman no. 42 filter paper and the filtrate was analyzed for residual fluoride concentration by SPADNS method, spectrophotometrically (UV-VIS spectrophotometer: Model No.Shimadzu UV 1240) at the wavelength of 570 nm described in the standard methods of examination of water and wastewater.

The percentage removal of the fluoride was calculated as follows:

$$\% \text{ Removal} = \frac{Ci - Ce}{Ci} \text{x} 100$$

Where, Ci is the initial fluoride concentration (mg/l),

Ce is the equilibrium concentration of fluoride solution (mg/l).

Understanding of adsorption technique is possible with knowledge of the optimal conditions, which would herald a better design and modeling process. Thus, the effect of some major parameters like effect of contact time, Initial fluoride concentration, p^H, dose of adsorbent and stirring rate were investigated. Adsorption studies were performed by batch technique to obtain the equilibrium data. All the experiments were conducted at room temperature (29 ± 2 °C).

a) Effect of Contact Time on fluoride removal efficiency.

Contact time plays a very important role in adsorption dynamics. The effect of contact time on adsorption of fluoride onto fly ash (TPP fly ash and Maize Husk fly ash) is shown in Figure15-24. Batch adsorption studies using the concentrations 3.0, 5.0 and 7.0 mg/l of fluoride solution and with 1.0 g of the adsorbent with particle size of 20-30 μm were carried out at constant stirring rate of 400 rpm.

b) Effect of variation of p^H

The p^H of the aqueous solution is a controlling factor in the adsorption process. Thus, the role of p^H at 2, 4, 6, 8, 10, 12 was observed. The p^H was maintained at desired value with ±0.2 by adding 0.5 N HNO_3 or 0.1 N NaOH with 50 ml of prepared solution of 7 mg/l of fluoride solution for contact time of 120 min with a dose of 1 g/50 ml of 20-30 μm particle size fly ash powder (TPP fly ash and Maize Husk fly ash).

c) Effect of Adsorbent dose on Adsorption Capacity and Efficiency

Studies on effect of adsorbent doses were conducted by varying adsorbent doses between 0.25 to 2.5 g/50 ml. The p^H was maintained at 7, while initial fluoride ion concentration was fixed at 7 mg/l and contact time was kept as 120 minutes. Stirring rate of 400 rpm was set to carry out the experimental work using the two adsorbents (TPP fly ash & MH fly ash).

d) Effect of Stirring rate on fluoride removal efficiency

Studies on the effect of stirring rate (rpm) on fluoride removal efficiency (percentage) were conducted by varying speeds from 50 rpm to 400 rpm by using the mechanical stirrers, at pH of 7(neutral) with adsorbent dose of 2 g/50 ml and contact time of 120 minutes. The initial fluoride concentration of the test solution/sample was taken as 7 mg/l.

e) Effect of Initial fluoride concentration on Adsorption Capacity and Efficiency

Studies on the effect of initial fluoride concentration were conducted by varying the concentration from 3 to 25 mg/l keeping adsorbent dose of 2.0 g/50 ml, pH of 7, stirring rate of 200 rpm (for TPP fly ash) and 250 rpm (for MH fly ash) and contact time of 120 minutes.

4.3 THE ADSORPTION ISOTHERMS

Four isotherm models were used to fit the experimental data namely: Langmuir model, Freundlich model, Temkin Model and Redlich- Perterson Model. Langmuir, Freundlich model, Temkin Model and Redlich-Perterson Model were chosen to describe the adsorption equilibrium.

Freundlich, Langmuir, Redlich-Perterson and Temkin isotherms were plotted to provide deep insight to the adsorption of fluoride on Thermal Power Plant fly ash and Maize husk fly ash. The isotherms not only provides the general idea of the effectiveness of the Thermal Power Plant fly ash and Maize husk fly ash in removing fluorides, but also indicates the maximum amount of fluoride ions that will be adsorbed by the Thermal Power Plant fly ash and Maize husk fly ash. However, adsorption isotherms are equilibrium tests and thus do not indicate the actual performance of the adsorbent.

Langmuir isotherm is valid for single-layer adsorption [51]. It is based on the assumption that all the adsorption sites have equal affinity for molecules of the adsorbate and there is no transmigration of adsorbate in the plane of the surface

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