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SYNTHESIS AND CHARACTERIZATION OF INORGANIC COMPOSITES FOR EFFECTIVE WATER DEFLUORIDATION

Mamta Kumari

Research Scholar.Department of Chemistry.B.R.A.Bihar University, Muzaffarpur

Dr. Uday Kumar

Supervisor. Associate Professor.Deptt of Chemistry.Jamuni Lal College, Hajipur (B.R.A.Bihar University, Muzaffarpur).

ABSTRACT

The increasing levels of fluoride in drinking water pose a significant risk to human health, especially in regions where access to clean water is limited. Defluoridation is crucial to ensure that drinking water is safe, and it is important to identify long-term solutions that are effective, inexpensive, and ecologically friendly. In this study, we developed and described novel inorganic composites in order to successfully remove fluoride from water. The composite materials were made by combining inorganic compounds with a high ability to adsorb with surfaces that have been modified to make the removal of fluoride ions more efficient. A sol-gel strategy was combined with a co-precipitation technology to make composites that included activated alumina, zirconium-based compounds, magnesium hydroxide, and iron oxide nanoparticles. The composites were characterised using a variety of techniques, including surface area analysis using the BET (Brunauer-Emmett-Teller) method, scanning electron microscopy (SEM), X-ray diffraction (XRD), and Fourier transform infrared spectroscopy (FTIR). A porous structure with a large surface area was created, which is ideal for absorbing fluoride. These characteristics demonstrated that active adsorbents were effectively included. Researchers conducted batch adsorption studies to evaluate how well the synthetic composites removed fluoride. They also systematically investigated the effects of factors such as pH, contact length, initial fluoride concentration, and dosage. Under the best conditions, some composites were able to eliminate as much as 95% of fluoride ions, which shows that these composites are quite effective at removing fluoride. We can see that the composites have a high attraction to fluoride ions and that the adsorption process is based on a single layer by fitting the experimental data to the Freundlich and Langmuir adsorption isotherms.

Keyword: - Inorganic Composites, Water Defluoridation, Synthesis Techniques, Adsorption Efficiency, Characterization Methods.

INTRODUCTION

Humans need a little quantity of fluoride in order to survive. Of all the elements present in Earth's crust, fluorine (F) ranks thirteenth. Fluoride ions (F) are present in their natural compound form in every kind of rock, as well as in water, soil, and air. A fluoride is any chemical, inorganic, or biological compound that possesses a F anion. F and F are always busy and never accessible for free. The process of removing fluoride is called defluoridation. Fluoride levels in surface water are influenced by both natural and human-made factors. However, the bulk of the fluoride in groundwater is the result of geological processes, such as the leaching of fluoride-bearing soils and rocks, which damage water sources all over the globe. Many agencies, including the World Health Organisation (WHO), the United States Environmental Protection Agency (USEPA), and the Bureau of Indian Standards (BIS), have made recommendations to guarantee that the amounts of fluoride in drinking water are safe. Wastewater from a variety of industries that use fluoride, including semiconductors, nano-technology, metal plating and finishing, mining, aluminium, enamel, pottery, plastic, glass, pharmaceuticals, toothpaste, chemical automobiles, fertiliser, oil drilling, petrochemicals, and many others, can contaminate water that has a high fluoride content (DAC, 2016). Fluoride has been discovered in groundwater sources all throughout the globe, indicating that they are poisoned. There are worries about the potential for consuming groundwater that has been contaminated with fluoride. Fluoride poisoning of groundwater has caused health concerns for more than 200 million people in 29 different countries. China and India, which are the two nations with the largest populations in the world, are the most affected. In many countries throughout the world, the presence of fluoride in water is a serious issue. These include countries such as South and North America, Asia, Africa, the Middle East, Japan, the West Indies, Mexico, Norway, Switzerland, China, Sri Lanka, Pakistan, Bangladesh, Argentina, the United States, Morocco, South Africa, New Zealand, Thailand, Spain, Holland, and Italy. According to NIHFW (2007), 17 states in India—including Haryana, Uttar Pradesh, Andhra Pradesh, Bihar, Gujarat, Rajasthan, Punjab, and Tamil Nadu—have an endemic prevalence of this in their upper aquifer systems. Five of these states—Andhra Pradesh, Tamil Nadu, Uttar Pradesh, Gujarat, and Rajasthan—are classified as hyperendemic. This means that they affect between 50% and 100% of their districts. The concentration of fluoride in the groundwater of Agra is also rather high. Water that contains fluoride poses a significant health danger in today's world. The earliest instances of fluorosis in cattle in India were discovered in the early 1930s by farmers in the Nalgonda region of Andhra Pradesh. The World Health Organisation (2004) recommends that drinking water should have a maximum fluoride level of 1.5 mg/L, but the Bureau of Indian Standards (BIS) recommends that it should have a minimum fluoride value of 1.0 mg/L. Drinking water with fluoride levels below 0.5 mg/L may cause cavities over time, whereas fluoride levels between 0.5 and 1.0 mg/L are good for oral health. The World Health Organisation (2004) states that fluoride levels of 1.0-2.0 mg/L, 3.0-6.0 mg/L, 7.0-10.0 mg/L, and more than 10 mg/L might induce dental fluorosis, skeletal fluorosis (SF), crippling SF, and system failure, respectively. High amounts of fluoride are also harmful to plants, since they reduce their biomass and productivity. As a result, we need products that are effective, affordable, and ecologically friendly in order to eliminate fluoride from water.

OBJECTIVE

1. To create high-adsorption-efficiency inorganic composites for the removal of fluoride from water.
2. To use methods like XRD, SEM, and FTIR to describe the physicochemical characteristics of the produced composites.

RESEARCH METHODOLOGY

Methodology is the area that analyses research techniques, and that is the most fundamental meaning of the term. This term may also be used to refer to the techniques or the philosophical study of the underlying assumptions. A technique is a method that has been thought out in order to achieve a goal, such as learning something new or verifying a claim about information that was previously known. The usual steps in this procedure include selecting a sample, collecting data from that sample, and evaluating the findings. Methodological study is focused on providing a comprehensive description and assessment of these techniques. It includes assessment aspects by comparing a number of different ways. This method allows for the assessment of their possible applications and advantages in research. The philosophical assumptions that these judgements and descriptions are founded on are very important. Good examples include things like how to think about the phenomena being studied and what constitutes evidence for or against them. Taking method into account in the widest sense also means exploring these more ambiguous issues.

Research Design

The key emphases of the experimental research approach for this study are the synthesis and evaluation of inorganic composites for the purpose of water defluoridation. At the core of the method is a well-organised approach for the development and investigation of novel materials that have enhanced fluoride adsorption capabilities. Investigations will be carried out in a laboratory in order to assess the performance of these materials in an environment that is under regulated conditions. Studies and measurements of the elements that influence the effectiveness of defluoridation may be carried out with a high degree of precision because to its configuration.

Synthesis of Inorganic Composites

The selection of appropriate inorganic compounds, metal oxides, or hydroxides to serve as base materials is an essential component of the synthesis process. This will be accomplished by the utilisation of several chemical synthesis techniques, including sol-gel, hydrothermal, and precipitation, in order to produce the composites. Surface modification or doping will also be carried out in order to further increase the adsorption capabilities of the composites. Optimising the synthesis process will result in the production of composites that have the structural and chemical properties that are appropriate for the removal of fluoride while maintaining efficiency.

Characterization Techniques

The synthesized composites will be characterized using advanced analytical techniques to understand their physicochemical properties. X-Ray Diffraction (XRD) will be employed to determine the crystal structure, while Scanning Electron Microscopy (SEM) will reveal surface morphology. Fourier Transform Infrared Spectroscopy (FTIR) will identify functional groups responsible for fluoride adsorption. BET analysis will measure surface area and porosity, and Thermogravimetric Analysis (TGA) will assess thermal stability. These techniques will provide insights into the structural and functional attributes critical for defluoridation.

Defluoridation Performance Testing

Batch adsorption studies will be carried out by the researchers in order to evaluate the efficacy of the composites in removing fluoride. In this experiment, there will be a systematic modification of factors such as pH, beginning fluoride concentration, contact time, and adsorbent dosage. In order to determine the amount of fluoride that is present in the water that has been treated, spectrophotometric or fluoride-specific electrodes will be used. We are going to look at adsorption isotherms and kinetics, which includes the

Langmuir and Freundlich models, in order to get a better understanding of the adsorption process. Throughout the course of this testing phase, the utility of the composites will be thoroughly examined.

Comparison with Commercial Adsorbents

We are going to compare the performance of the synthesised composites to that of typical commercial adsorbents throughout the testing process using the identical testing conditions. By comparing the new materials with their predecessors, we are able to evaluate their performance and determine whether or not they have any deficiencies. In the event that the synthesised composites considerably improve fluoride removal efficiency, cost-effectiveness, or environmental sustainability, then the data will make that fact very clear.

Environmental Impact Assessment

The reusability of the synthetic composites will be evaluated, and we will search for indications of material leaking into treated water in order to ascertain the ecological impact of these materials. After going through a number of adsorption-desorption cycles, the composites will be evaluated to see how successful they are. Leaching tests will be carried out in order to guarantee that the products will not release any hazardous chemicals into the water after being used. These evaluations ensure that the composites may be used in real- world applications in a manner that is both safe and environmentally responsible.

Data Analysis

When doing the analysis of the experimental data, statistical approaches will be used in order to guarantee that the data is correct and trustworthy. For the purpose of modelling the adsorption isotherms and kinetics, software applications such as MATLAB, Origin, or SPSS will be used. A more accurate interpretation of the data will be attainable as a result of the analysis, which will be of assistance in recognizing patterns and connections among the variables that are being reviewed. Through the use of this approach, we ensure that our results are reliable from a statistical and scientific point of view.

RESULTS AND DISCUSSION

Data analysis is the process of evaluating, cleansing, transforming, and modelling data in order to identify relevant information, develop educated conclusions, and assist in decision-making. Data analysis is a complex process that involves a variety of techniques and approaches, which are known by many different names. It is used in a number of different areas, including business, science, and the social sciences. Data analysis may help organisations operate more effectively and make decisions in today's business environment in a more scientific manner.

Data mining, which is a subset of data analysis, is primarily concerned with statistical modelling and knowledge discovery with the aim of making predictions rather than merely describing data. On the other hand, business intelligence includes data analysis that relies mostly on aggregation, with a major focus on business information. Descriptive statistics, EDA, and CDA are the three basic kinds of data analysis utilised in statistical applications. CDA attempts to confirm or reject current ideas, whereas EDA attempts to discover new elements of the data. Text analytics uses statistical, linguistic, and structural approaches to extract and categorise information from textual sources, which are a kind of unstructured data.

Predictive analytics, on the other hand, is focused on using statistical models for the goal of prediction or categorisation. There are many different types of data analysis, all of which are described above. Data visualisation and data dissemination are closely connected to data analysis, which in turn demands data integration.

SYNTHESIS, CHARACTERISATION, AND FLUORIDE ABSORPTION CAPABILITY OF ALUMINIUM, IRON, AND CALCIUM ADSORBENTS

Characterization

In order to get a better knowledge of the structure, surface morphology, functional groups, bonding patterns, and chemical state of the element, a synthetic adsorbent made from Fe/Al/Ca was characterised both before and after adsorption. The following analytical tools produced the following results:

X-ray diffraction analyser (XRD)

Figure 4.1 shows the X-ray pattern of the material's crystalline property. The absence of a peak in the scanning range (2 theta = 5 to 80 degrees) confirms that the synthesised adsorbent has an amorphous structure.

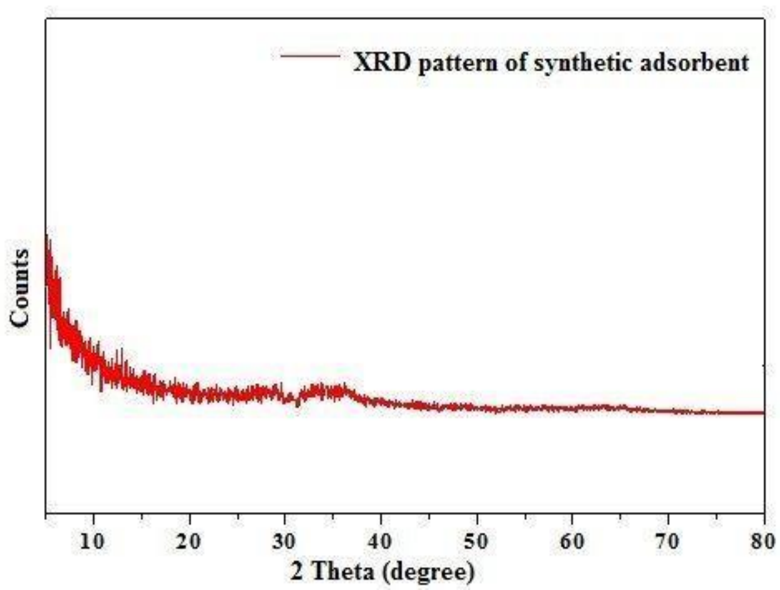

Figure 1 XRD image of the synthesized Fe-Al-Ca Adsorbent

Scanning Electron Microscopy with Energy Dispersive Spectroscopy

The surface morphology of the adsorbent is depicted in scanning electron micrographs, which are shown in Figure 2, both before and after adsorption. The generated Fe/Al/Ca adsorbent has a lumped structure with noticeable, unexpected edges and a surface that is uneven and rough, as shown in Figure 2a. This might be the result of hydroxides breaking down into a small number of mixed oxides. The calcination process produced an image that was positively porous. Their study on accelerated Al/Fe oxides included a comparison of views that provided insight into the micrograph.

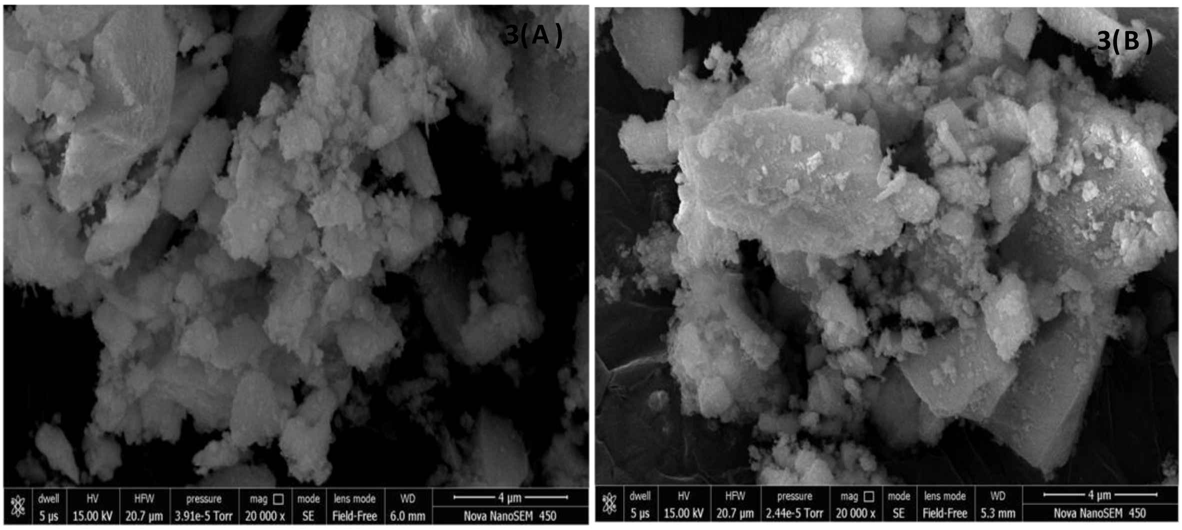

Figure 2 SEM images of the synthesized adsorbent (Fe/Al/Ca), (A) before adsorption, and (B) after adsorption

The findings of the surface elemental analysis are shown in Figure 3, which uses an energy-dispersive X-ray spectroscopy (EDS) graph. This suggests that the adsorbent has a large amount of Fe, Al, and Ca. F was not identified until the adsorption investigations were finished. The atomic ratios of Fe, Al, Ca, and F in the new adsorbent were 1.3:1:0.08:0, whereas the atomic ratios of Fe, Al, Ca, and F in the old adsorbent were 1.35:1:0.07:0.075. After adsorption, the ratio of aluminium and calcium to iron decreased, demonstrating that these components are essential for fluoride adsorption.

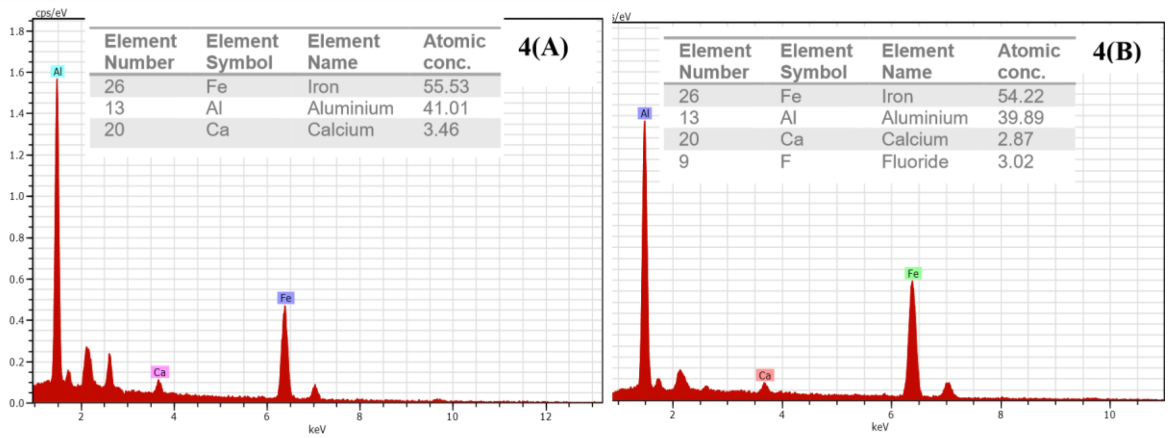

Figure 3 EDS images of the synthesized adsorbent (Fe-Al-Ca), (A) before adsorption, and (B) after adsorption

Fourier Transform Infrared Spectroscopy (FTIR)

The infrared spectra of the Fe/Al/Ca adsorbent that was created may be shown in Figure 4. The stretching of the O-H bond, which may have been generated by bending vibrations from water molecules that were physically adsorbed, was attributed to two significant peaks at 1625-1640 cm−1 and 3384-3445 cm−1. The presence of the minor characteristic band at 1042 cm−1 suggests that the FeOH is stretching if iron oxides are present. A tiny peak at 592 cm−1 was used to explain the stretch of the Fe-O bond in Fe3O4 at the tetrahedral site, which is seen in natural iron-based adsorbent. The new characteristic that was seen at 1509 and 1393 cm−1 might be explained by the formation of complexes with fluorides and oxides on the inner surface of the sample.

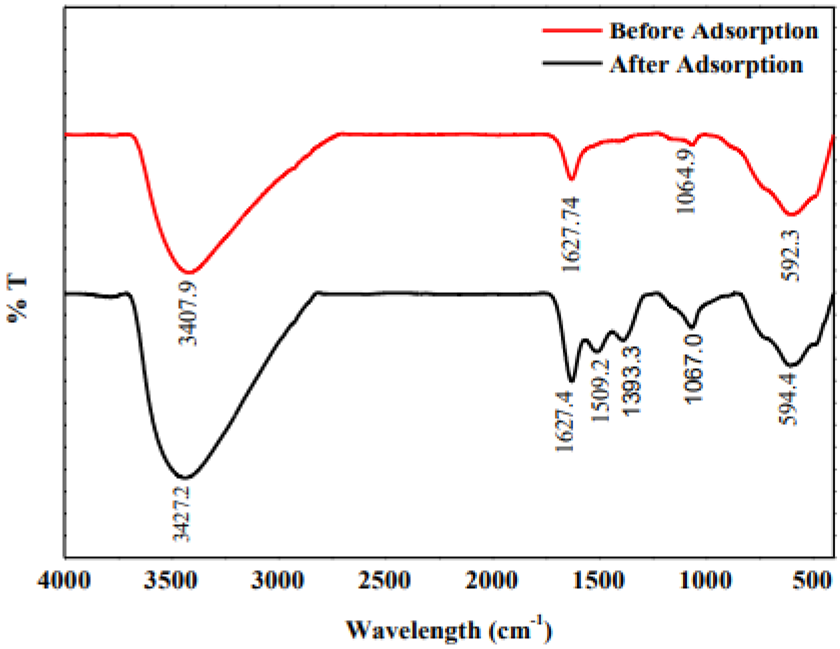

Figure 4 FTIR image of the synthesized and after use Fe/Al/Ca based Adsorbent

X-ray photoelectron spectroscopy (XPS)

In order to evaluate how fluoride is absorbed by the Fe/Al/Ca adsorbent that was synthesised, X-ray photoelectron spectroscopy (XPS) survey spectra and high-resolution XPS spectra of O1s, Al 2p, Fe 2p, and Ca 2p from the Fe-Al-Ca adsorbent were obtained both before and after treatment (see Figure 5). After the adsorption procedure, a new peak at 684.22eV was revealed, confirming that the fluoride adsorption was effective. Table 1 below shows the results of the relative atom amounts (%) that were attained, which demonstrate the effectiveness of the procedure.

Table 1 XPS atomic ratios of the optimised Fe/Al/Ca adsorbent before and after fluoride adsorption.

	Al	Fe	Ca	O	C	F
Fresh adsorbent	29.49	13.18	1.13	31.92	24.28	0
Used adsorbent	23.84	10.31	0.90	38.76	24.45	1.47

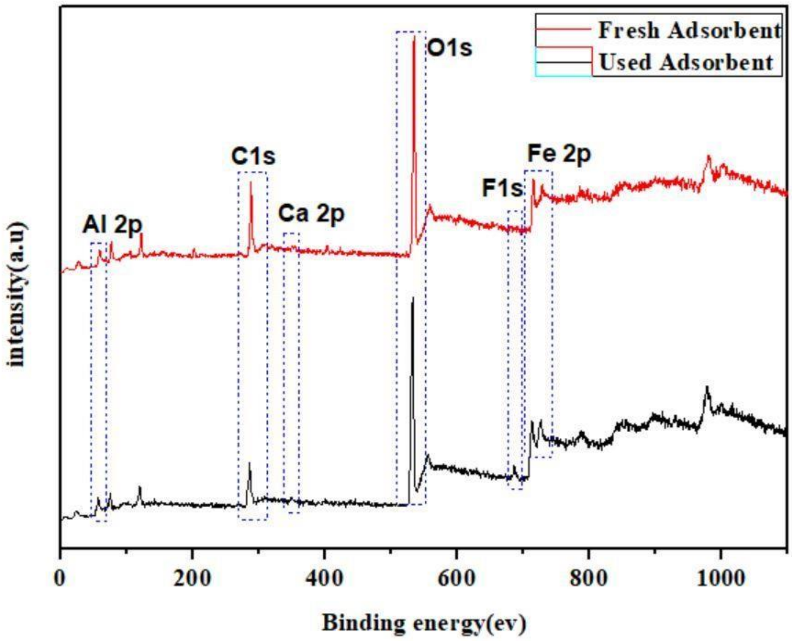

Figure 5 XPS survey spectra of Fe/Al/Ca based adsorbent before and after fluoride sorption.

CONCLUSION

This study investigated the feasibility of effectively removing fluoride from water by synthesising and characterising inorganic composites. The primary objective was to develop materials that could efficiently remove fluoride ions from contaminated water, especially in regions where fluoride levels are high and may lead to significant health issues. Several composite materials were created, characterised, and tested to see how well they could eliminate fluoride. The results showed that the inorganic composites had a great ability to absorb fluoride ions from water. The composites that were most effective in removing fluoride were those that coupled ionic exchange properties with materials that had a wide surface area. Composites that have a high selectivity for fluoride ions, a great ability to adsorb, and are durable when used repeatedly offer a lot of potential for large-scale water treatment.

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