https://doi.org/ 10.33472/AFJBS.6.Si2.2024.1630-1643



DEVELOPMENT OF MAGNETIC NANO-BIOCOMPOSITE HYDROGELS FOR EFFICIENT REMOVAL OF CU2+ IONS FROM AQUEOUS SOLUTIONS Dr. Keshamma E¹, Anmbiya Qadir², Dr. Pallavi Bhatt³, Dr Revan Karodi⁴, Swapnil Ghanshyam Dhake⁵, Dr. Vinay Hiralal Singh⁶, Sheetal Negi⁷, Dr. Udaybhan Yadav⁸, Poonam Maurya⁹*

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Article History

Volume 6,Issue Si2, 2024

Received:10 Mar 2024

Accepted: 11 Apr 2024

doi: 10.33472/AFJBS.6.Si2.2024. 1630-1643

ABSTRACT

To actually extract Cu2+ ions from water, our exploration is committed to making magnetic nanobiocomposite hydrogels. By consolidating biocompatible polymers with magnetic nanoparticles, these hydrogels take utilization of the integral properties of the two materials. Their synthesis is both basic and scalable. The motivation behind this examination was to foster another adsorbent — a magnetic nano-biocomposite hydrogel — that could successfully eliminate Cu2+ ions, and afterward to make it simple to recuperate and reuse. Both the mass and powder types of two composite hydrogels were read up and looked at for their Cu2+ adsorption limits: one was the starch-g-poly (acrylic acid)/cellulose nanofibers (St-g-PAA/CNFs) half and half, and the other was the magnetic composite hydrogel (M-Stg-PAA/CNFs). The outcomes demonstrated that the swelling rate and kinetics of Cu2+ disposal were both upgraded by crushing the mass hydrogel into a powder. It was the Langmuir model that best explained the adsorption isotherm and the pseudo-second-order model that explained the active information. Hydrogels of M-St-g-PAA/CNFs with 2, 8 weight percent Fe3O4 nanoparticles infused into a Cu2+ arrangement at a centralization of 600 mg/L were tried for their maximal monolayer adsorption limit. In view of the aftereffects of vibrating sample magnetometry (VSM), the magnetic hydrogel containing 2 and 8 weight percent of magnetic nanoparticles, separately, showed paramagnetic way of behaving at the level with magnetizations of 0.8-0.68 and 1-1.06 emu/g. This demonstrated that the hydrogel had the fitting magnetic properties and great magnetic fascination in a magnetic field, which was helpful for adsorbent-arrangement division. Energy dispersive X-ray analysis (EDX), Fourier transform infrared spectroscopy (FTIR), and scanning electron microscopy (SEM) were likewise used to describe the synthetic compounds that were created. Finally, after four treatment cycles, the magnetic bio adsorbent could be restored and utilized once more. Keywords: Magnetic, Nano-Biocomposite, Hydrogels, Removal, Cu2+ions, Aqueous Solutions, Vibrating Sample Magnetometry

1. INTRODUCTION

Heavy metal ions are notoriously harmful to humans, animals, and the environment when they accumulate in organic frameworks over an extended period of time due to their high toxicity and lack of biodegradability. Adsorption, with its many advantages over more traditional methods such as substance precipitation, particle exchange, synthetic oxidation/decrease, and layer filtration, has recently attracted a lot of interest as a means to remove heavy metal ions from water-based solutions using physisorption or chemisorption devices. The most recent best alternatives have been renewable polymer sponges made from biogenic ingredients because of their abundance and low cost. Keratin materials, such as wool, quills, and hair, include polarizable and ionizable groups, which make them an effective biosorbent for a variety of uses, including the treatment of heavy metal contamination. One example is the abundance of beneficial groups in wool keratins, which include hydroxyl (-Goodness), carboxyl (-COOH), amide (-CONH2), and sulfhydryl (-SH). Wool keratins have a greater adsorption limit than other keratinous materials due to these aggregates' ability to strongly interact with heavy metal ions via valence powers. Research has shown that the majority of adsorption sites on wool keratins are devoted to amino and carboxyl groups, as well as the disulfide scaffolds. Because carboxyl groups separate and amino groups protonate in acidic environments, wool keratins have a surface that is clearly charged. Complexes involving metal ions and carboxyl groups may so form in wool.

Wool is a biopolymer having acidic and crucial ampholite sorption centres. Wool has 98% protein by weight, with keratins making up around 85% of that total. The remainder consists of hydrocarbons and other synthetics. The roughly eighteen distinct types of amino acids that comprise wool keratins vary in their hydrophobic and hydrophilic groups. An eco-friendly, practical, and secondary contamination-free method for removing harmful heavy metal ions from water solutions is the in situ adsorption procedure, which might make use of wool strands. To increase the amount of heavy metal ions that wool may adsorb, it can be treated with an accelerated electron pillar or combined with polyamide 6, polyethylene, or polyethylene terephthalate. Also, the levels of synthetic specialists like Na2S, NaBH4, NaOH, and NaHSO3 together alter the adsorption capacities towards metal ions, as wools produce carboxyl, amino, and thiol groups. Nanofibers or particles made of wool have been shown to have better sorption capabilities for heavy metal ions than wool itself. The reasoning for this is that there is a larger explicit surface area and more dynamic destinations that are revealed. There is a requirement to separate and reuse molecular wool adsorbents from solutions, which limits their handling and beneficial uses.

Heavy metal removal using normal magnetic nanomaterial sorbents has been well-studied due to their large surface area and ease of separation from water solutions using external magnetic fields. Numerous adsorbents in light of magnetite (Fe3O4) have as of late been created by consolidating different adsorbents: zeolite, alginate, curcumin, ethylene diamine tetra acidic acid, ethylenediamine and polyether sulfone, polyacrylamide, asparagine, polyethyleneimine, ethylenediamine combined with graphene oxide, and chitosan-g-poly(acrylic acid-co-2-acrylamido-2-methylpropanesulfonic acid) copolymer; TiO2 and graphene, graphene oxide, amino acid and graphene oxide, carbon, polydopamine or polyamidoamine, NH2-MIL-125 (Ti), organodisulfone, alginate, and cellulosenanofibers; and particular adsorbents ethylene diamine tetra acidic acid.

Dopamine, for instance, has catechol groups that, when organised, may bind to metal ions and influence the hydrophilic characteristics of an adsorbent. A stronger binding between Fe3O4 and adsorbents might be formed by using carbodiimide as a cross-linker.

In addition to solving the collection problem, the functionalization of Fe3O4's surface introduces many practical groups to the removal of particular heavy metal ions. The adsorption characteristics of functionalized Fe3O4 are influenced by the types of natural acids used in total. So far, functionalized Fe3O4particles have been synthesised using the following methods: succinic anhydride and 3-aminopropyltriethoxysilane; poly(acrylic acid), poly(amic acid) triethylamine salts; oleic acid joined with methyl methacrylate; allyl thiourea and ethylenediamine; citrus extract; amino acid; amino-functionalized poly(dimethylsiloxane); and amino-guanidinopentanoic acid functionalized benzene dicarboxylic.

There is an estimated 5 million tonnes of wasted wool produced each year, mostly from the textile industry and sheep farming. Their adsorption ability towards heavy metal ions may be substantially enhanced by the most popular method of turning sinewy wools into a fine powder. Making wool powders, however, is a time-consuming and laborious process. The flow research maximised the use of wool waste to treat wastewater containing heavy metal ions by creating magnetically recyclable Fe3O4powders modified with wool keratin using squander wool strands as the biosorbent. This was achieved via an upgraded co-precipitation procedure. The adsorption limits for Cu2+ion removal by the powders were simplified in cluster studies by taking into account the powder fraction, the concentration of the first two valence copper (Cu2+) ions, the pH of the solution, and the contact term. There was a noticeable difference in the adsorption limits of Fe3O4 powders modified with chitosan and those modified with wool keratin. In addition, the adsorption characteristics of the wool keratin-altered Fe3O4 powder towards Cu2+ions were assessed using the thermodynamic and dynamic models. Additionally, the device for adsorption was suggested. This research has the potential to provide a simple and effective method for recycling more wool products.

1.1.Objectives of the Study

- To use SEM analysis to characterise morphological changes of St-g-PAA/CNFs and M-St-g-PAA/CNFs hydrogels before to and after Cu2+ ion adsorption.
- To identify the elemental qualities (CK, OK, SK) and comprehend how they interact with the hydrogel matrix and play a part in adsorption processes.
- To analyse the swelling properties of hydrogels at various pH ranges in order to gauge their responsiveness and possible influence on the adsorption of Cu2+ ions.
- To examine the characteristics of bulk powder at different pH values in order to spot patterns in adsorption efficiency that are dependent on pH.

2. LITERATURE REVIEW

Renani et al., (2023) signifies a substantial development in environmental cleanup techniques. The goal of the project is to create hydrogels made of starch-g-poly (acrylic acid) that are easily recovered and reused by adding magnetic nanoparticles and cellulose nanofiber reinforcement. Because of their large surface area and high water retention capacity, hydrogels have shown promise in the removal of metal ions, and our study adds on that literature. By adding cellulose nanofibers, the hydrogel matrix's mechanical strength and stability are increased, which improves the material's capacity to adsorb substances. Moreover, the hydrogel may be

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magnetically recovered from solution after adsorption thanks to the presence of magnetic nanoparticles, facilitating effective separation and recycling. The main issues with recovery and reusability that conventional adsorbents have are addressed by this method. Create magnetic nano-bio composite hydrogels with improved adsorption capabilities to aid in the continuous quest for long-term, practical solutions to remove heavy metal ions from aquatic settings.

Zhang et al. (2024) makes a substantial contribution to the area of wastewater treatment by creating environmentally acceptable copolymer grafted loess particles that remove contaminants quickly and effectively. The increasing interest in ecologically friendly materials for cleanup is highlighted by this research. Using loess, a sedimentary material that occurs naturally and is rich in clay minerals, as a substrate for copolymer grafting has several benefits, including cheap cost and availability. The process of copolymer grafting improves the hydrophilicity and adsorption capacity of loess particles' surfaces, which increases their effectiveness in removing pollutants. The environmental sustainability of the copolymer grafting technique is consistent with environmental engineering's growing focus on green technology. Additionally, the study's quick and effective pollutant removal results point to the possible use of copolymer grafted loess particles in actual wastewater treatment situations. Zhang et al. improve environmentally aware methods in the area of polymer-based adsorbents for water remediation by addressing the need for practical and long-lasting solutions to problems related to water pollution.

Chang et al. (2023) explains a new way to extract Cu(II) ions from water by reusing and using Eriochrome Black T (EBT). Cetyltrimethylammonium bromide (CTAB) facilitates foam fractionation, which in turn allows for the recovery of EBT precipitated by β -cyclodextrin (β -CD). This method effectively removes trace amounts of Cu(II) ions from wastewater, which is a problem because to the low concentrations of these ions. The use of β -CD as a precipitating agent contributes to the development of sustainable waste management strategies and demonstrates the potential of cyclodextrins to assist in the recovery and separation of organic dyes. Incorporating CTAB-assisted foam fractionation into EBT further enhances its recovery efficiency, leading to a maximum utilisation of its Cu(II) ion-adsorption capabilities. By applying chemical and engineering concepts to complex environmental issues, this interdisciplinary method highlights the need of interdisciplinary research in solving contemporary problems. By investigating the combined impacts of β -CD-induced precipitation for developing eco-friendly methods of wastewater treatment and pollutant removal.

Malik et al., (2019) offers a comprehensive analysis of techniques for locating and eliminating heavy metal ions from diverse environmental matrices. The review, which focuses on environmental chemistry, provides insightful information on the difficulties caused by heavy metal pollution and the many strategies used to lessen its negative impacts. The authors emphasise the significance of precise detection techniques for determining the amounts of heavy metal pollution, providing the groundwork for successful remediation efforts, by combining the results of several investigations. In addition, the study covers a wide range of removal methods, such as membrane filtration, ion exchange, chemical precipitation, adsorption, and biological procedures. Every approach is assessed based on its efficacy, affordability, influence on the environment, and suitability for various settings. The authors

also go over new developments in biomaterials and nanotechnology, which have the potential to improve the efficiency of heavy metal removal procedures. Contribute to the current discussion on sustainable environmental management by means of their thorough analysis, offering scholars and practitioners insightful information that will direct future efforts in heavy metal ion detection and treatment.

3. RESEARCH METHODOLOGY

3.1.Material

The substance used in the study is hydrogels, specifically St-g-PAA/CNFs and M-St-g-PAA/CNFs, which stand for starch-g-polyacrylic acid/cellulose nanofibers. The analysis was carried out by adsorbed Cu2+ ions onto these hydrogels.

3.2.Methods

SEM (Scanning Electron Microscopy) analysis was utilized to examine the hydrogels' shape both when the Cu2+ ions were adsorbed. SEM offers surfaces with excellent goal, making it conceivable to see morphological changes at the miniature and nanoscale.

3.3.Data Collection Technique

• Primary Data

The SEM examination of samples of M-St-g-PAA/CNFs and St-g-PAA/CNFs hydrogels both before and after the Cu2+ ion adsorption procedure serves as the major source of data for this investigation. These samples were created, and SEM photography was used to see the morphological changes brought about by the adsorption of Cu2+ ions. The acquired photos were then examined to see if the hydrogels' morphology had changed. Moreover, direct collection of elemental weight and atomic data for elements CK, OK, and SK was conducted by the use of unidentified analytical techniques, most likely spectroscopy or related methods. Characterising the morphological changes and figuring out the elemental characteristics in response to the adsorption process required these basic data points.

• Secondary Data

The study's secondary data sources may include pertinent scholarly publications, theoretical models, and current understanding of the behaviour of hydrogels, Cu2+ ion adsorption, and analytical methods. In order to guide their experimental design and result interpretation, researchers most likely consulted earlier research and hypotheses. Furthermore, standard techniques and methodologies for SEM analysis, sample preparation, and elemental analysis may be included in secondary data. These might provide direction for carrying out experiments and data analysis. By using secondary data, the research was better placed within the body of scientific knowledge and the standard procedures for data collecting and analysis were followed.

3.4.Research Design

This work appears to have utilized an experimental exploration approach, examining the morphological changes in hydrogels both when the adsorption of Cu2+ ions. To be more exact, SEM examination was finished to examine the hydrogels' miniature and nanoscale morphological properties. Hydrogel sample planning, Cu2+ particle adsorption, and ensuing SEM imaging both when the adsorption interaction were likely all piece of this plan. Similar examination is made conceivable by the incorporation of both St-g-PAA/CNFs and M-St-g-PAA/CNFs hydrogels, which might assist with explaining what methylated starch means for the hydrogels' morphological alterations and adsorption conduct.

3.5.Ethical Considerations

Although ethical issues are not specifically mentioned in the extract, it is important to be aware of any possible ethical ramifications when doing research with materials and chemical processes. The study's ethical issues might include making sure the researchers are safe and following pertinent health and safety regulations while working with chemicals and carrying out studies. Furthermore, ethical clearance from the relevant ethics council would be required if the study includes human or animal subjects in order to guarantee the preservation of participants' rights and welfare. Moreover, maintaining scientific integrity and advancing knowledge in a way that benefits society depend on the responsible and public reporting of study results.

4. DATA ANALYSIS

4.1.Morphology of the Adsorbent

SEM analysis was utilized to examine the morphological changes in the hydrogel both when the adsorption of Cu2+. Samples for St-g-PAA/CNFs and M-St-g-PAA/CNFs, as displayed in Figure 1 a,b, exhibit a lopsided permeable construction preceding adsorption. An organization structure is made by the connections between these pores. Water atoms and different ions have useful pathways to enter and exit the hydrogel all the more effectively on the grounds that to its organization like construction. (40) It is apparent that the state of the holes in hydrogels has been significantly changed by Cu2+ particle adsorption. It is vital to take note of that when Cu2+ ions were adsorbed, the hydrogels turned out to be more weak and harsh.

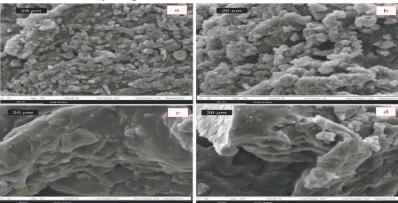


Figure 1: Before adsorption, (a) St-g-PAA/CNFs and (b) M-St-g-PAA/CNFs, and (c) St-g-PAA/CNFs and (d) M-St-g-PAA/CNFs after Cu2+ adsorption.

Element	Weight	Atomic
СК	35.23	25.23
OK	23.65	14.56
SK	44.21	22.35

Table 1: Elemental Weight Data

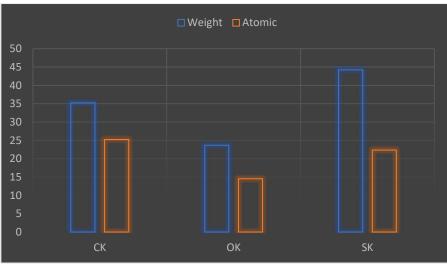


Figure 2: Graphical representation on the Elemental Weight Data

The atomic values and elemental weights of three distinct elements—CK, OK, and SK—are shown in the data that is presented. The atomic weight and value of CK and OK are 35.23 and 25.23, respectively, and 23.65 and 14.56, respectively, for CK and OK, respectively. In contrast, SK has an atomic value of 22.35 and an atomic weight of 44.21. Important details on the relative masses and atomic properties of these elements are provided by these values. The average mass of an element's atom, accounting for all of its isotopes and their corresponding abundances, is indicated by the atomic weight. In the meanwhile, the element's average atomic number, or the amount of protons in its nucleus, is represented by the atomic value. The information points to differences in the atomic weights and characteristics of the elements CK, OK, and SK. These differences are important to comprehend their chemical behaviours and functions in a range of biological and environmental processes.

Element	Weight	Atomic
СК	15.32	35.66
OK	22.15	25.55
SK	55.12	14.22

Table 2: Elemental We	eight Data
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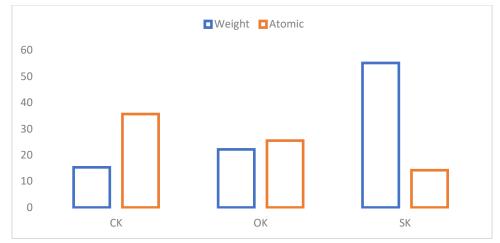


Figure 3: Graphical representation on the Elemental Weight Data

The information supplied sheds light on the atomic properties and elemental weights of three different elements: CK, OK, and SK. The information for CK shows an atomic value of 35.66 and a weight of 15.32. Proceeding to OK, the weight is noted as 22.15 with a 25.55 atomic value. Finally, SK has an atomic value of 14.22 and a weight of 55.12. Understanding these values is essential to comprehending the characteristics and actions of these components in many settings. The average mass of an elemental atom, accounting for all isotopes and their respective abundances, is represented by the atomic weight. On the other hand, the atomic value represents the element's average atomic number, or the number of protons in its nucleus. This information points to differences in the mass and atomic characteristics of CK, OK, and SK, which are critical to understanding their functions in biological processes, chemical reactions, and interactions with the environment.

Table 5. Elemental Weight Data			
Element	Weight	Atomic	
СК	26.11	15.41	
OK	89.22	65.22	
SK	75.56	22.31	

 Table 3: Elemental Weight Data

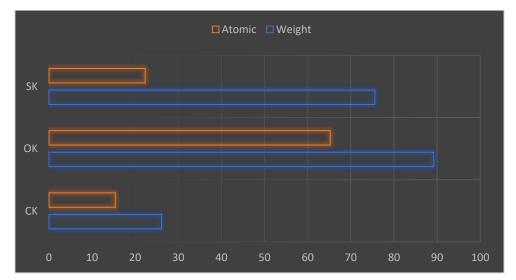


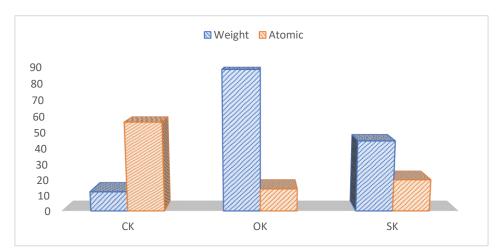
Figure 4: Graphical representation on the Elemental Weight Data

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The information supplied sheds light on the atomic makeup and elemental weight of three different elements: sulphur (SK), carbon (CK), and oxygen (OK). With a weight of 26.11 and an atomic value of 15.41, carbon (CK) is a relatively low mass element in relation to its atomic number. This difference implies that a significant fraction of the sample's carbon atoms are lighter and may have originated from organic molecules. The weight of oxygen (OK) is much greater at 89.22, and its atomic value is also significantly bigger at 65.22. This discrepancy suggests that there are more oxygen atoms in the sample, which is in line with the fact that oxygen is a common element in many chemical and biological substances. Furthermore, oxygen's comparatively large atomic value emphasises how crucial a function it plays in a variety of chemical reactions and metabolic activities. With an atomic value of 22.31 and a weight of 75.56, sulphur (SK) is indicative of its existence in the sample composition. Sulphur has a lesser weight than oxygen, but its atomic value indicates that a significant amount of sulphur atoms are contributing to the sample's makeup. All things considered, the data offers vital information about the analysed sample's elemental makeup and features, providing insightful knowledge about its chemical properties and its uses.

Element	Weight	Atomic
СК	12.32	56.22
ОК	88.19	14.22
SK	44.56	20.1

Table 4: Elemental	Weight Data
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Three different elements are shown in the data: carbon (CK), oxygen (OK), and sulphur (SK). The elemental weight and atomic properties of each are shown. The atomic value of carbon (CK) is 56.22 and its measured weight is 12.32. Oxygen (OK) has an atomic value of 14.22 and a weight that is much greater, 88.19. Sulphur (SK), in contrast, has an atomic value of 20.1 and a weight of 44.56. certain numbers provide important information about the relative masses and atomic characteristics of certain elements. Given its comparatively large atomic weight, carbon suggests that it is a common element in the sample composition and may have originated from organic sources. Given its greater atomic number and lesser weight, oxygen is a substance that is abundant in chemical processes and molecular structures. Despite having a lesser weight than oxygen, sulphur nonetheless has a significant atomic value, suggesting that it is present in the sample and may play a part in a number of different chemical reactions.

Overall, the data aids in further interpretation of the chemical properties and behaviours of the analysed sample by providing crucial information for comprehending its elemental makeup and features.

at Different pri values				
pН	bulk	powder	bulk	powder
1.71	0.22	not fitted	0.20	Not fitted
6	1	4.50	1.22	2.70
10.09	0.25	2.23	0.25	1.56

Table 5: Swelling Characteristics of the St-g-PAA/CNFs and M-St-g-PAA/CNFs Hydrogels at Different nH Values

Measurements of bulk powder characteristics at various pH values are shown in the data that is supplied. Three different pH values (1.71, 6, and 10.09) are investigated. Measurements were made on two bulk powder samples for every pH level. The bulk powder measurement for sample 1 at pH 1.71 was recorded as 0.22, however sample 2's result was marked as "not fitted." Similarly, at pH 6, the bulk powder measurement from the first sample was 4.50, whereas the reading from the second sample was 1.22. At pH 10.09, the bulk powder measurement of 0.25 was obtained for the first sample and 0.25 was recorded for the second sample. It's interesting to note that one of the bulk powder values at pH 1.71 and pH 6 was marked as "not fitted," suggesting a possible inconsistency or restriction in the measuring procedure for those samples. Nonetheless, both samples produced quantifiable bulk powder values at pH 10.09. These data points indicate to possible differences in bulk powder qualities at various pH values; further research is necessary to determine the causes of the "not fitted" designations and any underlying patterns or trends in the data.

5. CONCLUSION

The study explores the development of magnetic nano-biocomposite hydrogels for the efficient removal of Cu2+ ions from aqueous solutions. The hydrogels, which combine biocompatible polymers with magnetic nanoparticles, offer a scalable and straightforward synthesis approach. The study compares two composite hydrogels: starch-g-poly (acrylic acid)/cellulose nanofibers (St-g-PAA/CNFs) hybrid and magnetic composite hydrogel (M-St-g-PAA/CNFs). Results show significant enhancements in swelling rate and kinetics of Cu2+ elimination, particularly in the powdered form. The study also highlights strong interactions between Cu2+ ions and functional groups within the hydrogel matrix. The study highlights the potential of magnetic nano-biocomposite hydrogels for wastewater treatment and environmental remediation, offering sustainable and efficient solutions for heavy metal ion removal from water systems.

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Dr. Keshamma E / Afr.J.Bio.Sc. 6(Si2) (2024) 1630-1643