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Original Research Article

Synthesis and application of Tin Ferrite (SnFe2O4) as photocatalyst for the degradation of Ochratoxin A(OTA) in aqueous solution

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ABSTRACT

Ochratoxin A (OTA), one of the frequently detected mycotoxins in food and other environmental matrices, requires sequestration to enhance the environment and human health. This preliminary study emphasizes the screening of photocatalysts for the degradation of OTA in an aqueous environment via the preparation, characterization, and photocatalytic activity of SnFe2O4 material. SnFe2O4 was synthesized through the co-precipitation process, and surface and structural characteristics were carried out via Scanning Electron Microscopy connected Energy Dispersive X-ray (EDX) spectroscopy, Electron Mapping, and Fourier Transform Infrared (FTIR) spectrophotometric analysis. SEM analysis indicates a uniform agglomeration of tiny particles that develop into an octahedral structure, representing the morphological formation of SnO. FTIR analysis further confirmed the availability of functional groups Fe-O, Sn-O, and O-Sn-O at 400-500 cm-1, 590 cm-1, and 1194 cm−1on the surface of the SnFe2O4 photocatalyst's spectrum. EDX gave insight into the elemental composition, confirming the availability of Fe, Sn, and O with varying percentage weight (wt. %) of 33.7, 32.9, 29.9, and 3.2, respectively, in the synthesized photocatalyst. At the same time, electron mapping also showed their distribution contained in the Sn ferrite for regularity towards achieving better photocatalytic activity. Sequestration study towards degradation of OTA in aqueous solution attained 100 % removal in the dark after about 40 min. However, a haphazard removal pattern was observed during the study under irradiation under simulated visible light. This study indicates the potential of SnFe2O4 proficient material for the removal of OTA in an aqueous environment, providing an environmentally friendly technique towards the detoxification of mycotoxin-water.

Keywords: Ochratoxin A; Detoxification; Photocatalysis; Sn Ferrite; Mycotoxin

1.0 INTRODUCTION

Mycotoxins are secondary intermediates generated by fungi, which pose a substantial global health concern due to their widespread contamination of food and feed. These toxic compounds are generated by different fungal species such as Aspergillus, Penicillium, Fusarium, and Alternaria, and can inflict severe harmful

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effects on vertebrates (Awuchi et al., 2021; Oliveira et al., 2025). The term "mycotoxin" itself is derived from "mykes" (fungus) and "toxic" (poison), aptly describing these chemical products formed by fungi that readily colonize crops in the field or post-harvest (Yvv et al., 2016). The global economic losses attributed to mycotoxin contamination are substantial, impacting food safety and international trade due to their pervasive presence in agricultural commodities (Cinar and Onbaşı, 2020). Beyond farm products, an increasing apprehension vis-à-vis the likelihood of mycotoxin contamination in drinking water sources keeps intensifying, for an underexplored avenue for human exposure and health risks. These mycotoxins are present in water sources such as drinking water sources (Oliveira et al., 2018; Koko et al., 2024) and bottled water (Mata et al., 2015). Mycotoxins are detected in minimal concentrations, but their continuous accumulation could be detrimental to human health. For instance, Oliveira and colleagues reported a concentration of about 35 ng.L-1 of Aflatoxins B1 and B2, fumonisin B3, and ochratoxin A (OTA) in some drinking water sources (Oliveira et al., 2018), while Koko and coauthors reported mean concentrations of 14.96 ± 4.46, 8.59 ± 3.86, and 10.56 ± 2.84 $\mu g.L^{-1}$ for zearalenone, OTA, and deoxynivalenol in some drinking water sources in three southwestern States in Nigeria (Koko et al., 2024). The mycotoxin, OTA is known to pose several severe impacts on different organs/systems in living organisms which include kidney, liver, nervous system, embryo (foetus) and immune system on numerous animal species (Malir et al., 2016) and have been categorized as Group 2B (probable human carcinogen), because of the verified carcinogenicity in animal experiment by the International Agency for Research on Cancer (IARC) (El Khoury and Atoui, 2010, Bui-Klimke and Wu, 2015). The IARC has also identified OTA as a potential human carcinogen due to its consideration as a significantly dangerous nutritional risk factor (Cagnasso et al., 2019). OTA remains stable in humans, with a t½ of 35 days in blood subsequent to a single oral dose owing to the difficulty in its toxicokinetic elimination (Petzinger and Ziegler, 2000). The European Food Safety Authority (EFSA) established a bearable weekly ingestion of 0.12 µgkg-1 body weight (bw). The prevalence of this mycotoxin in the aquatic environment necessitates the imperative need for its removal to prevent its impact on organisms and human beings, considering its occurrence in drinking water sources.

Detoxification via the adoption of physical (solvent extraction, irradiation, and high temperature heating), chemical (acidification, alkalization, and ozone treatment), and biological techniques (microbial degradation) remain the primary means for neutralizing mycotoxins in different matrices (Jing et al., 2024). These methods have been reported to pose some threats with several challenges accrued to them, such as resistance, expensive equipment required, production of numerous byproducts, utilization of a large amount of solvent, etc. Other researchers have worked to curb the challenges through the adoption of other techniques, such as adsorption,

for instance, in a recent study by Yang and Colleagues on developing an effective and facile technique for the uptake of multiple mycotoxins in an aqueous environment. The authors were able to engineer a novel material, a nanofiber mat improved with polydopamine and ionic liquid (PDA-IL NFsM) that successfully absorbed and concurrently eliminated numerous mycotoxins in an aqueous environment and achieved high elimination efficacy >88.3 % for the majority of the 15 mycotoxins among seven classes of the mycotoxins (Jin et al., 2023). Despite the advantages, adsorption as a technique has been known to exhibit some challenges, hence the need to adopt new and practical strategies for sequestering these mycotoxins in water. Photocatalysis in different phases has become an optimistic, cost-effective, green/eco-friendly, and easily applicable method capable of completely breaking down organic and inorganic related contaminants to less toxic compounds like CO_2 and H_2O (Ameta et al., 2018; Zhang et al., 2018). Although the technique seems compelling enough to obtain a photocatalyst that is equally content with experimental conditions to enhance efficacy, like thermal conditions optimization, photostability, and thermal stability, it remains a serious problem. Therefore, the provision of material to meet the required standard originates from the material fabrication to the application (Dostanić, Lončarević et al. 2024). With a dearth of information on the occurrence of mycotoxins in aquatic systems, little has been reported on the photocatalytic breakdown of mycotoxins in water. Several photocatalysts have been adopted for the degradation different mycotoxins in food, La–ZnFe2O4@Fe3O4@carbon (98.37 % of AFB1), patulin (97.35 %) and zearalenone (98.52 %), Fe3O4@carbon (69.57 %) and $ZnFe_2O_4$@Fe_3O_4@carbon (91.39 %) (Yang et al., 2023); 2-AP-grafted ultrathin (AFB1 (100%) and ZEN (89.5 %) (Wang et al., 2019); metal-organic framework material, Zirconium-Metalloporphyrin (PCN-222(Mn)) (OTA (90 %)) (Zhu et al., 2025), graphene/ZnO (ZEN (99 %)) (Bai et al., 2017); TiO2 (AFB1(94 %), ZEA (100 %), and OTA (97 %) (Sousa, 2017).

Recently, the use of ferrites (MFe_2O_4) has gained attention in their use for the breakdown of organic contaminants in water owing to their small band-gap (about 2.0 eV, which has significant photosensitivity to visible light), magnetic property, and high stability (Sharma et al., 2013). Additionally, the magnetic and optical properties of Fe_3O_4 (simplest ferrite) could be easily altered by substituting Fe^{2+} ions with other bivalent cations (Mn, Co, Ni, Cu, Zn, Ca, and Mg) (Wu and Song, 2023; Gupta et al., 2020). Among these, Sn ferrites have been considered very promising in the photocatalytic applications in different fields owing to their attractive, unique functional properties such as better chemical stability, great electrical resistivity, excellent biocompatibility, earth-abundant ability, and excellent recoverability due to their magnetic capability (Han et al., 2022); (Elmoussaoui et al., 2012). $SnFe_2O_4$ is known to be a p-type semiconductor that possesses a small energy bandgap (~2.53 eV), which permits the absorption of visible light within the electromagnetic spectrum (Han et al., 2022). $SnFe_2O_4$ and their composites have found

exciting applications in the sequestration of different organic contaminants, $SnFe_2O_4$ (treatment of cancerous (A549) human cell lines) (Sargazi et al., 2021), sulfur-doped $SnFe_2O_4$/graphene (chlorotetracycline) (Jia et al., 2017); $SnFe_2O_4$ nano-octahedron on Au (CO2 reduction and chlorotetracycline degradation) (Li et al., 2022); $SnFe_2O_4$/$ZnFe_2O_4$ heterojunctions (tetracycline) (Wang et al., 2020); $SnFe_2O_4$/SnO_2/PANI (methylene blue and methyl orange) (Arshadnia et al., 2017); $SnFe_2O_4$ decorated on bentonite substrate (amoxycillin) (Fatolahi and Maleki, 2024); $SnFe_2O_4$@activated carbon magnetic nanocomposite (crystal violet) (Rai et al., 2015), and acrylamide on magnetic $SnFe_2O_4$/CeO_2photocatalyst was utilized to degrade some dyes(Aboelfetoh et al., 2024). Tin ferrite has been an excellent photocatalyst for the degradation of some organic contaminants in different matrices, and could serve as a better material for the breakdown of OTA in water due to its advantages, including magnetic property, enabling easy removal from the system after the treatment process. This is because this photocatalyst has not been used for the degradation of these mycotoxins in the water environment. Hence, in this study, SnFe2O4 nanoparticle was synthesized using the co-precipitation method using $SnCl_2.2H_2O$ precursor and $FeCl_3{\cdot}6H_2O$ precursor with NaOH as a reducing agent. The structural and morphological composition of the material was examined using FT-IR spectroscopy, SEM, electron mapping, and EDX. A photocatalysis simulation experiment was done to examine the degradation of OTA within water.

2.0 Materials and Methods

2.1 Materials and reagents

$FeCl_3.6H_2O$ (98%), $SnCl_2.2H_2O$ (99.8%), NaOH (98%), Ethylene glycol (99.5%), Citric acid (99%), methanol, and Sodium acetate (99%) were obtained at Glass World (Glassblown, volumetric Glassware and Chemicals, South Africa). Distilled H_2O used was generated using the ELGA Purelab Flex Water Purification System, Company, Department of Chemical Engineering, University of Pretoria, South Africa. Ochratoxin A (OTA) in acetonitrile Oekanal (34037-2ML-R) was purchased from Merck Life Sciences (Pty) Ltd, Modderfontein, 1609, South Africa. Stock solution contains 10 µg/mL of OTA in 2 mL of acetonitrile, a calibration curve from the stock was prepared, 0.01, 0.04, 0.05, 0.1, 0.2, 0.3, 0.4, and 0.5 µg. mL-1 that was used for quantification.

2.2 Methodology

2.2.1 Synthesis of $SnFe_2O_4$

Stoichiometric amounts of tin chloride ($SnCl_2{\cdot}2H_2O$) and of ferric chloride ($FeCl_3{\cdot}6H_2O$) were combined in 200 mL of methanol and agitated for 0.5 h. The pH of the suspension was estimated to be 0.70. After which, 200 mL of 1.0 M NaOH(aq) was introduced dropwise until the pH of the solution was obtained to be 13.0. Formation of precipitates was observed, after which it was separated by filtration to detach the salts and unreacted components in the system. The precipitation was afterwards placed in an oven at 80 °C for 12 h to obtain a moisture-free photocatalyst. The as-prepared material was

calcined in a muffle furnace at 550 °C and ground, kept in an airtight bottle for further analysis.

2.2.2 Characterization of SnFe2O4

The morphology and microstructure of the samples were observed by a SEM (Hitachi-S4800) coupled with energy-dispersive X-ray (EDX) spectroscopy. Fourier transform infrared spectroscopy (FT-IR) was recorded using a Nicolet Avatar 370 FT-IR to determine the functional groups contained in the prepared material.

2.2.3 Photocatalytic studies

The simulated light source was generated with a 216 W (4 × 54 W) Fluora fluorescent lamp with a combined light strength of approximately 183 mW/cm2. The light source was placed at a distance of 0.1 m from the conical flask placed on the stirrer. In the conical flask, 0.025 g of the material was introduced to 10 mL of OTA solution (0.1 µgmL-1). Before the irradiation, the solution was continuously agitated without light for 0.5 h to attain an adsorption–desorption equilibrium. Within that period, an aliquot of the stirring samples was collected at a time interval of 10 min to determine the removal efficiency of OTA within the dark experiment. Subsequently, when the light was introduced, about 1 mL of solution was sampled periodically, every 10 min, and the experiment was run for 240 min to assess the disparity of OTA concentration at different times. The aliquots were filtered using a 0.22 µm syringe filter to eliminate particles of the photocatalyst contained in the sampled solution and were later analyzed with an Alliance Waters 2695 separation Module HPLC-UV detector. OTA was analyzed at the wavelength of 333nm, A = Methanol, and B = methanol, column temperature = 40 °C, column = C18, long length, injection volume = 10 µL, flow rate = 1 mL/min, and total run time = 5 min.

The percentage removal [RE (%)] was evaluated using the formula below (Eq. (1)):

$$\%\, removal = \frac{(Co - Ct)}{Co} x100 \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots (1)$$

Where Co is the initial amount of aqueous OTA; Ct is the amount of aqueous OTA at the irradiation time (t).

4.0 Results and Discussions

4.1 Characterization of $SnFe_2O_4$

IR spectroscopy provides information on the functional groups contained in compounds, attributed to the stretching and vibration bands identified in the spectrum, as presented in Figure 1. The two absorption areas observed are associated with Sn-O and Fe-O bonds seen at spectral 400-500 and 590 cm^{-1}, respectively, found in the crystal structure (Shokri et al., 2018). The vibrations within 882–1222 cm^{-1} designate the existence of the bond C-O from the alcohol group; the absorption band of 1384 cm^{-1} indicates the availability of hydroxyl functional groups (O–H) resulting from alcohols. A vibration frequency peak of Sn-Fe was observed at 1384 cm^{-1}, while that at 1194 cm^{-1} indicates the O-Sn-O bond contained in the synthesized material (Adewuyi et al., 2023). The absorption area between 1590–1690 cm^{-1} demonstrates bending vibrations owing to the existence of Fe-OH bonds, consequential from the partial oxidation of Fe2+ to Fe^{3+} during preparation. A good stretching

double band at 2358 cm^{-1} shows the availability of carbon dioxide (O = C = O) (Arshadnia et al., 2017), because of exposure to the atmosphere. A thin absorption peak at 2991 cm^{-1} was detected, due to the C-H bond stretching, resulting from a straight-chain hydrocarbon. The absorption band between 3600-3740 cm^{-1} is due to H-bonds, showing the existence of moisture (H_2O) within the sample (Sedaghati-Jamalabad and Bagheri-Mohagheghi, 2024).

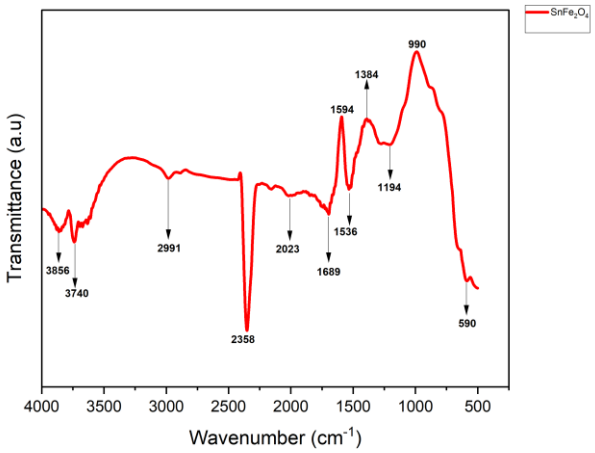

Figure 1: FTIR spectrum of $SnFe_2O_4$

The typical scanning electron micrograph of $SnFe_2O_4$ is shown in Figure 2, indicating a uniform agglomeration of tiny particles that form an octahedral structure, representing the morphological formation of SnO (Etminan et al., 2018). The chemical composition and elemental distribution on the surface of the synthesized material were determined using SEM coupled with EDX/mapping; the micrograph is shown in Figure 3. The prepared material revealed the presence of elements with varying percentage weight (wt. %) of 33.7, 32.9, 29.9, and 3.2 for Fe, O, Sn, and Na, respectively. Characteristic peaks were observed in the EDX spectrum of $SnFe_2O_4$ related to Sn, Fe, O, and Na with intensity inferred from the composition of the element contained in the as-prepared material (Jafari et al., 2021). The presence of a small amount of Na in the material could be attributed to its utilization as a reducing agent in the co-precipitation preparation method.

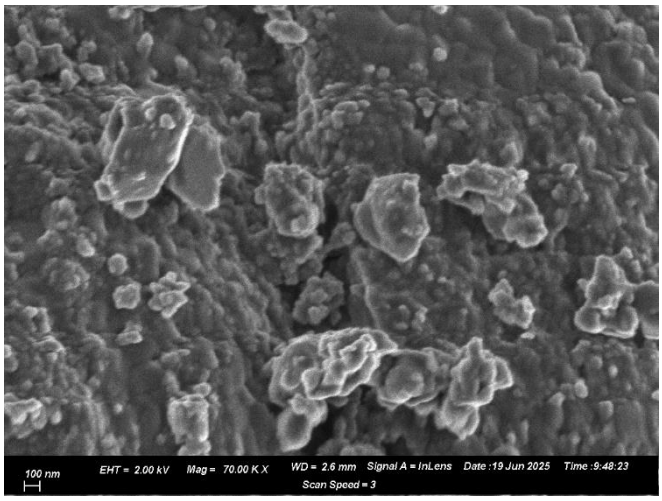

Figure 2: Scanning electron micrograph of $SnFe_2O_4$.

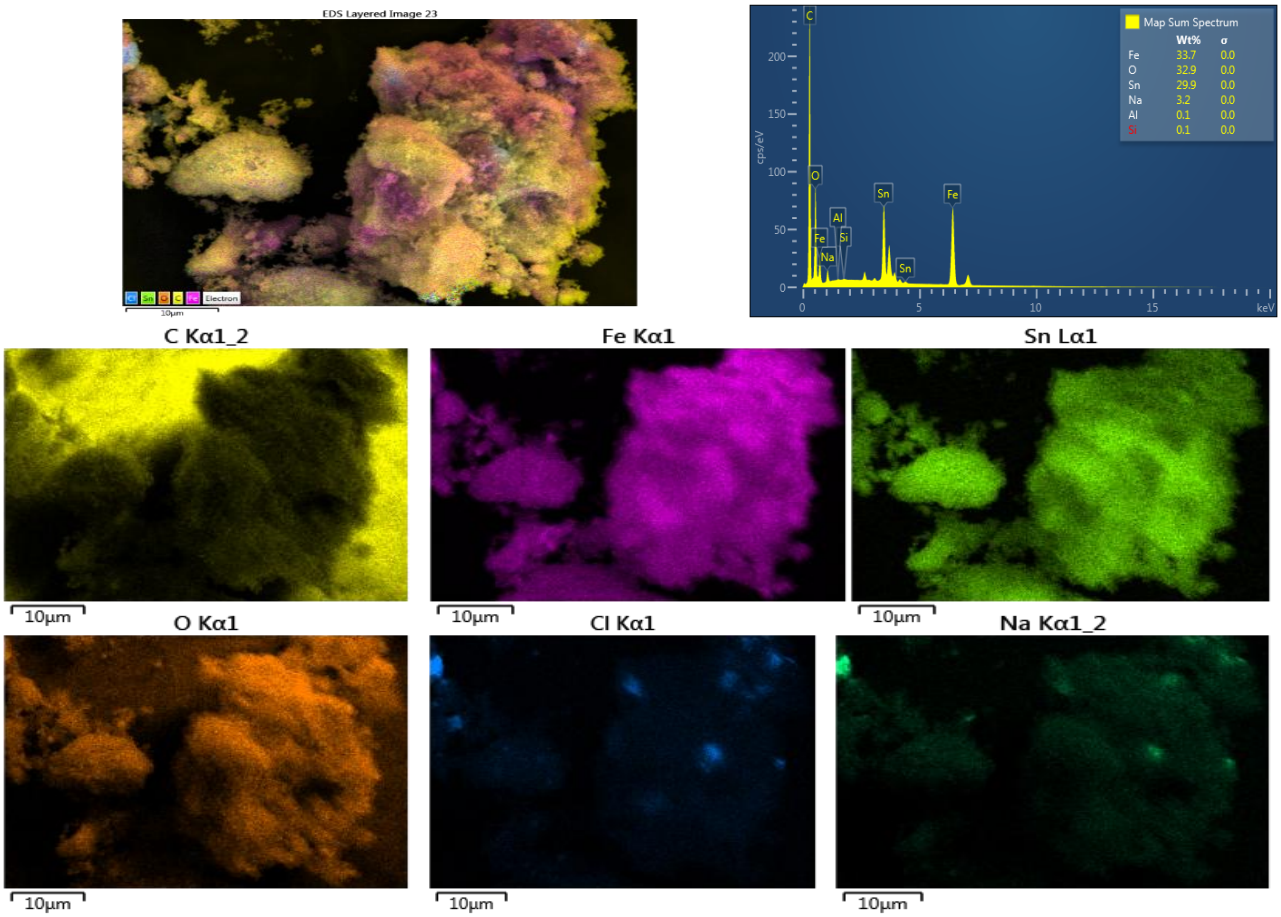

Figure 3: Electron mapping and EDX spectra of $SnFe_2O_4$.

4.2 Photocatalytic degradation of OTA by $SnFe_2O_4$.

$SnFe_2O_4$ and their nanocomposites have been found to have good photocatalytic capability on the degradation of some organic compounds (Kaur et al., 2023; Sanchez-Lievanos et al., 2024), including sulfamethoxazole, ciprofloxacin, erythromycin, and ampicillin (Adewuyi et al., 2023), diclofenac (Abilarasu et al., 2021), Congo Red and Acetophenone (Iqbal et al., 2019), Dye Degradation and Nitrophenol Reduction (Singh and Singh, 2024), in an aqueous environment. The photocatalytic activity of the tin ferrite material was examined based on its capability to break down organic contaminants in visible light. In this study, for removing mycotoxins (OTA), simulated visible light was used for their degradation in an aqueous environment. The results obtained from the kinetic study show the effects of the synthesized material on mycotoxins, with varying degrees of percentage removal, as shown in Figures 4 and 5. The percentage removal under dark conditions achieved a maximum removal capacity of 100 % with a period of 30 min, with gradual removal at different period intervals, 0 to 30 min, of the mycotoxin solution interaction with the synthesized material.

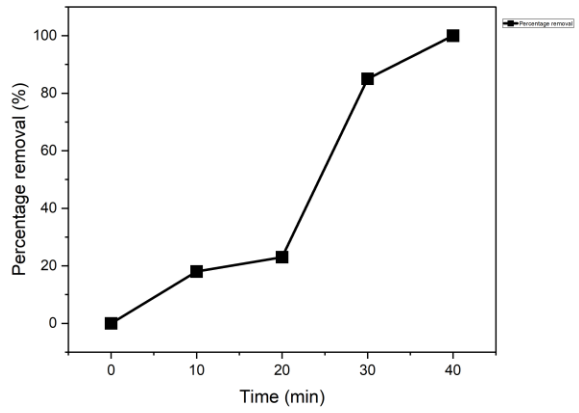

Figure 4: Removal percentage from the dark experiment

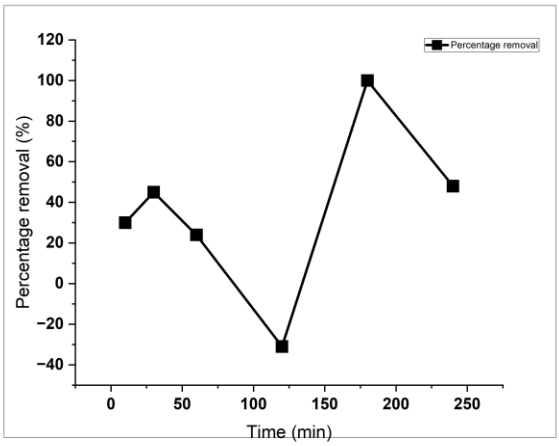

Figure 5: Percentage removal under light irradiation

From dark experiment, kinetic studies have shown gradual degradation that reached 85 % after 30 min of interaction of $SnFe_2O_4$ with OTA solution within the aqueous environment (Figure 4), which reached a maximum of 100 % within 0 min of irradiation light. This could be attributed to the fact that adsorption at the first phase, known as the fast stage, within the period of 0-30 min (Song et al., 2022). Under irradiation with simulated visible light, the removal increased gradually from 30 – 45 % from 10 min to 30 min, then decelerated from that period to 24 % after 120 min. The percentage removal then surged to completion (100%) after 180 min of irradiation, but later decreased to 48 % after 240 min. Photocatalytic study for removing OTA in water has shown inconsistency in removal, which could be attributed to simultaneous adsorption-desorption occurring during irradiation under simulated visible light. Also, the inconsistency removal could result from the fact that some photocatalysts can facilitate reactions in the dark via the adsorption of organic contaminants and enhance their breakdown through reduction/oxidation reaction. This insinuates that the material functions in the dark, facilitated by surface-active sites (Kumar et al., 2020; Ghasemi et al., 2016). The complete sequestration in the dark experiment has indicated a favorable phenomenon, such as adsorption, which could happen in the absence/presence of a light source (Hariri and Dehghanpour, 2021).

5.0 Conclusion

Ochratoxin A (OTA) degradation using $SnFe_2O_4$ proves to be a promising method for tackling this carcinogenic mycotoxin's toxicity and environmental effects. Material has been synthesized and characterized using SEM, FTIR, and EDX, which confirmed the composition of the elements/functional groups contained in the synthesized photocatalyst. The photocatalytic characteristics of SnFe2O4 enable the removal of OTA, which could be achieved effectively under precise conditions, creating an essential technique for detoxifying polluted surroundings.

The preliminary study has shown a maximum removal percentage of less than 60 min, suggesting adsorption as a possible process for removing OTA. In contrast, photocatalytic evaluation, on the other hand, has recorded a random removal pattern, which could result from the breakdown of the compound into other byproducts. The influence of various factors, like pH, photocatalyst dosage, temperature, and mechanism, could be studied to improve their removal efficiency as well as the byproducts generated during irradiation under light sources. As an alternative to traditional detoxification techniques, $SnFe_2O_4$ is a feasible and sustainable offering, a possible route to decrease the dangers posed by OTA contamination in water sources.

Author contributions

DTK., AAB., and HUH performed the experiment, literature search, data analysis, and drafted the manuscript. MOO and EIU conceptualized the study and critically revised the work.

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Compliance with ethical standards

Conflicts of interest: The authors have no relevant financial or non-financial interests to disclose.

Consent to participate: All authors declare their consent to participate.

Consent for publication: All authors declare their consent to publish the article.

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