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Activated Carbons Derived from *Areca Catechu* for the Removal of Aflatoxin in Aqueous Media

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ABSTRACT: Agricultural and food industry by-products, commonly known as biowastes, have emerged as valuable resources for developing highly porous carbon materials, particularly in the production of high-performance activated carbon (AC). These AC materials have proven their versatility across numerous separation and purification applications. This study explores the potential of Areca catechu husk-derived AC for aflatoxin (AFs) removal from aqueous solutions. The synthesis process incorporated chemical activation using orthophosphoric acid followed by thermal treatment at 500°C for 2 h. The resulting material exhibited exceptional characteristics, including well-defined porous structures spanning multiple size ranges and abundant oxygen-containing functional groups. These features contributed to its impressive surface area (699.53 m^2/g) and substantial pore volume (1.03 cm^3/g). Fluorescence spectrometry was used to evaluate the performance of the AC by monitoring the changes in AFs B_1 concentration of a 60 ppb aqueous solution over a 5 min period, providing rapid analytical detection well-suited for the fast removal kinetics observed. The results showed high adsorption capacity as evidenced by rapid AFs removal, with 0.1 g of adsorbent completely adsorbing 3.0 µg of AFB_1 within 120 s, which provides a substantial time advantage over previously studies. At the same time, the AC can be easily stored and used while requiring no pH adjustments to the treatment solution. These findings suggest that Areca catechu husk-based AC represents a promising alternative adsorbent material, particularly for targeting organic contaminants in aqueous environments.

Keywords: activated carbon, sustainable development goals (SDGs), *Areca catechu*, aflatoxin, organic pollutants

1. INTRODUCTION

Aflatoxins (AFs) represent a significant class of toxic secondary metabolites produced naturally by certain *aspergillus* species, particularly *A. flavus* and *A. parasiticus*. Among the four major AFs variants, B_1 (AFB_1), B_2 (AFB_2), G_1 (AFG_1) and G_2 (AFG_2), AFB_1 is considered the most potent and widely studied. The International Agency for Research on Cancer (IARC) has designated these compounds as Group 1 carcinogens, indicating potential severe health implications to humans. The prevalence of AFs contamination in Southeast Asia reaches 57%, second only to fumonisins at 85%, only further cements AFs contamination as a nationwide issue. However, AFs pose a more serious threat due to their Group 1 carcinogenic classification, while other mycotoxins typically fall into less severe categories (Group 2B or 3).[1] The above fact likely explains why the scientific community has focused more heavily on AFs in previous studies, despite the widespread occurrence of other mycotoxins in food supplies. The severity of AFs exposure cannot be understated. One notable historical aflatoxicosis outbreak has occurred in Malaysia, namely in 1988 that resulted in 13 paediatric deaths in Perak.[2] Prevention of future aflatoxicosis cases warrants further research into reliable, efficient ways of removing AFs from the environment.[3]

The classification of AFs as emerging contaminants (ECs) reflects the growing global concern towards their carcinogenic properties, particularly their impact on liver health. These compounds frequently contaminate various agricultural products, including groundnuts, maize and chilli peppers, through fungal invasion during cultivation or storage.[4] On the other hand, recent studies have also revealed the presence of AFs in various water sources, including surface water, drinking water and industrial effluents.[5] Their widespread presence raises significant health concerns, as chronic exposure has been linked to liver cancer and nutritional disorders such as kwashiorkor and developmental retardation, while acute exposure can trigger aflatoxicosis, resulting in liver failure and eventually, death.[6] The challenge of detecting AFs in water systems lies in their lack of obvious sensory indicators as they are odourless and tasteless. Additionally, their resistance to common degradation methods such as heat means that they cannot be degraded via conventional boiling or cooking methods at home.[2]

Contaminants of emerging concern (CECs) such as AFs typically cannot be removed through conventional wastewater treatment methods due to their polar or semi-polar nature.[7] Thus, AFs removal strategies to address this issue generally fall into two categories: degradation and adsorption.

Degradation methods break down AFs molecules into less toxic molecules, which generally involves an attack on either the furofuran, lactone or difuran rings.[8] Common degradation approaches include UV irradiation (up to 98% AFB_1 removal within 90 min), visible light photocatalysis using metal catalysts, ozonation (38.96% maximum

AFB_1 degradation) and biological treatment using enzymes such as laccases and peroxidases (90%–100% removal of major AFs). Care must be taken to ensure that the degradation products are not harmful to human health through careful evaluation involving cytotoxic tests. Additional limitations include longer treatment times (30 min–100 min for irradiation methods, several hours to days for biological methods), potential metal contamination from photocatalysts and the need for specialised equipment such as UV lamps.[9]

Adsorption methods, on the other hand, physically bind AFs to the surface of a material known as an adsorbent. Unlike degradation approaches that chemically alter AFs structure, adsorption methods rely on physical interactions between the adsorbent and the AFs molecule for efficient removal of AFs from solution. Some common adsorbents include mineral clays (bentonite, montmorillonite achieving up to 98% removal), plant-based biosorbents (durian peels showing 98% efficiency, grape pomace reaching 82% within 15 min) and carbon-based materials. Carbon-based adsorbents have demonstrated particularly impressive performance, with carbon xerogel microspheres achieving 100% removal within 15 min.[9] An example of a widely used carbon-based adsorbent is AC, which shall be the focus of this study. The surface of an AC has numerous, tiny pores that increase the surface area available for adsorption of contaminants. Since AFs molecules are physically removed and adsorbed onto the AC surface, degradation products will not be produced.

Most commercial ACs for water treatment are derived from non-renewable petroleum sources.[10] The use of these non-renewable sources raises concerns about supply limitations, cost and the release of toxic residues into the environment. Many reported methods also rely on expensive catalysts or plant extracts, reducing feasibility for large-scale application.[9] There remain knowledge gaps around the degradation mechanisms and by-products of various AF removal strategies. Much of the existing research focuses solely on removing AFB_1 at ppm concentrations, while AFs can occur in lower ppb levels in contaminated wastewater and crops, comprised of various AFs analogues.[11] The non-specificity and non-degradable nature of AC adsorbents has also been cited as a potential limitation.[12] Finally, most reported removal methods are intended for food and feed matrices, with limited study in aqueous systems. There is a need for more sustainable, economical and effective methods to remove the complex mixtures of AFs found in wastewater at environmentally relevant trace levels. Additionally, further research on renewable, widely available resources as precursors of AC for the removal of AFs are needed.

It was proposed that the synthesis of *Areca catechu* AC for the removal and detection of AFs from agricultural products and food to address this significant public health issue. *Areca catechu*, commonly known as betel nut palm, is a species of palm abundant across tropical regions of Asia including Malaysia. While the nut is associated with its

pharmacological and therapeutic benefits, its consumption is associated with many adverse health effects, which include an increased risk of oral cancer.[13–16] On the other hand, inedible components such as the husk and fronds, are typically disposed of as agricultural waste.[17] This presents an opportunity to find beneficial applications for what is otherwise squandered biomass. The development of AC from agricultural waste such as *Areca catechu* husks aligns with the United Nations Sustainable Development Goals (SDGs), particularly SDG 13 (Climate Action) through sustainable resource utilisation reducing environmental burden, SDG 6 (Clean Water and Sanitation) through water treatment applications, and SDG 12 (Responsible Consumption and Production) through circular economy utilisation of agricultural by-products.

Conversion of *Areca catechu* husks into AC holds promise considering its lignocellulosic composition.[18] According to previous literature, ACs derived from biowaste have demonstrated high adsorption capacities and efficiencies for removing various contaminants in water.[19] Hence, in this study, *Areca catechu* was selected as a precursor for AC production as it is known for its high carbon content and availability as an agricultural by-product.[20,21]

2. EXPERIMENTAL

2.1 Chemicals and Materials

Fresh, mature and dry husks of *Areca catechu* were sampled and obtained from trees in the vicinity of Sultan Mahmud Airport in Terengganu's coastal region. The following chemical reagents, all purchased from Sigma Aldrich, were used in their original form: AFB_1 (3 ppm, 1 mL ampule), orthophosphoric acid and Millipore-filtered water as the solvent.

2.2 Procedure for the Production of AC

The production of AC in this study was based on a previously established method with modification.[22] Dried *Areca catechu* husks were carefully compressed into a 200 mL beaker. Then, 200 mL of phosphoric acid (5 M) was added to achieve a 1:1 impregnation ratio. The mixture was then left undisturbed in a fume chamber for 24 h. The next day, the sample was transferred to an oven (1,200°C, Horiba, Japan) to undergo thermal activation at 500°C with a constant heating rate of 20°C/min for 2 h under inert nitrogen atmosphere. Following thermal activation, the sample was left to cool at room temperature for 24 h. The resulting product then underwent repeated and extensive washing cycles until the desired neutral pH condition of the eluate was achieved by monitoring using litmus paper. Vacuum filtration, followed

by overnight dehydration at 120°C and finally careful mechanical reduction using a mortar and pestle to achieve uniform particle size distribution, yielded the final AC product which was stored and sealed in an airtight Falcon tube.

2.3 Characterisation Methods

The surface morphology of the AC was examined using a scanning electron microscope (SEM, Tescan Vega, Czech Republic) operated at an accelerating voltage of 10 kV–15 kV with a working distance of ~10 mm. High-resolution transmission electron microscopy (HR-Transmission electron micrograph ([TEM], JEOL JEM-2100Plus, Japan) was employed at 200 kV to further investigate the textural properties and lattice structure of the AC.[22]

Elemental composition was analysed by SEM equipped with energy-dispersive X-ray spectroscopy (SEM-EDX). Fourier-transform infrared (FTIR, IR-Tracer100, Japan) spectra were recorded on a Shimadzu IRTracer-100 spectrometer in the range of 4,000 cm^{-1}–401 cm^{-1} with a resolution of 4 cm^{-1}, averaging 32 scans, to identify functional groups present on the AC surface.[22]The specific surface area and pore size distribution were determined by nitrogen adsorption–desorption at 77 K using the Brunauer–Emmett–Teller ([BET], Micromeritics, USA) method. Prior to analysis, the samples were degassed under vacuum at 150°C for 6 h. Surface area was calculated by the BET equation, while pore size distribution was evaluated using the Barrett–Joyner–Halenda (BJH) model. [22]

2.4 Evaluation and Optimisation of AFs Removal

A working solution of 60 ppb AFB_1 working solution was prepared by diluting the stock solution with deionised water. AC (0.1 g) was added to the solution under magnetic stirring at 120 rpm. Samples were collected at 1 min intervals over 5 min and filtered using syringes. AFs concentrations were measured using fluorescence spectrometry (excitation: 360 nm, emission: 440 nm) with reference to a calibration curve. The amount of AFs removed from the test solution with respect to time is an indication of the removal efficiency of the prepared AC. A plot of AFs fluorescence intensity against time was used to evaluate the optimal removal time of AF using AC.

3. RESULTS AND DISCUSSION

3.1 FTIR Analysis

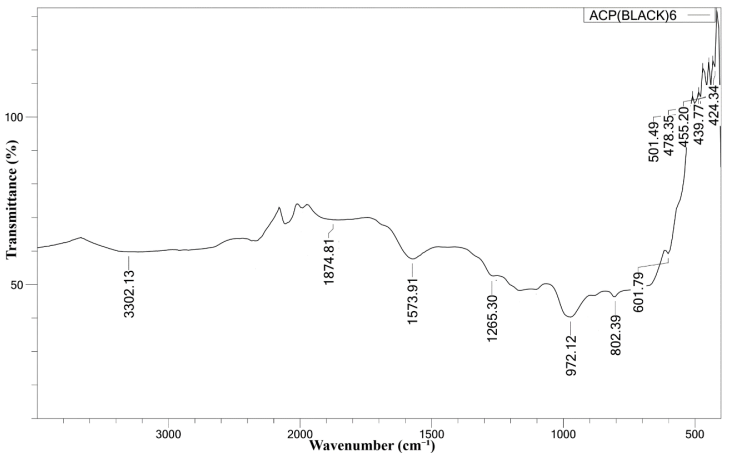


Figure 1: FTIR spectrum of AC from *Areca catechu*.

The FTIR spectrum of the AC revealed characteristic functional groups on its surface. From Figure 1, the absorption band at 3,300 cm^{-1} corresponds to O-H stretching vibrations from surface-bound water molecules.[23] These oxygenated functional groups which originate from the lignocellulosic substrate are believed to enhance the material's adsorption capacity by providing additional binding sites for AFs molecules.[24]

Strong bands observed at 1,874 cm^{-1} and 1,851 cm^{-1} indicate carbonyl stretching vibrations from organic components in the starting material. Additional bands at 1,573 cm^{-1} and 1,265 cm^{-1} represent aromatic C=C stretching and symmetric C-H deformation typical of cellulose and hemicellulose structures. The region between 1,000 cm^{-1}–1,165 cm^{-1} and 900 cm^{-1}–650 cm^{-1} showed characteristic stretching frequencies of C-O, C-C and C-O-C bonds, confirming the presence of cellulosic components. These FTIR results have revealed that lignin, cellulose and hemicellulose were the main contributors to the functional groups present in the AC.[25]

3.2 Thermogravimetric and XRD Analysis

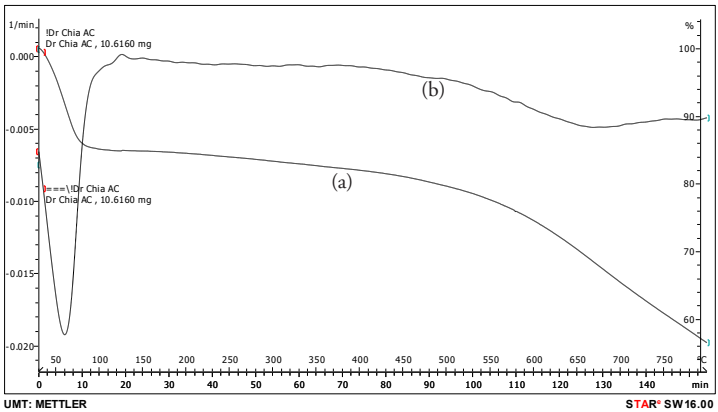


Figure 2: Thermogravimetric (TGA) curves of the prepared AC. (a) TGA curve and (b) differential (DTG) curve

Thermogravimetric (TGA) analysis was performed from 25°C–800°C under nitrogen atmosphere. Figure 2 shows three distinct regions in the thermal decomposition profile: evaporation of physi-orbed water or moisture (30°C–80°C, 15% weight loss), stability region with little mass change (80°C –500°C, 5% weight loss) and volatile matter release (above 500°C, 25% weight loss). The thermal profile showed minimal mass loss up to 500°C, suggesting that major structural changes and pore network formation occur below this temperature. This observation supports our selection of 500°C as the carbonisation temperature for achieving optimal pore development while minimising carbon loss.

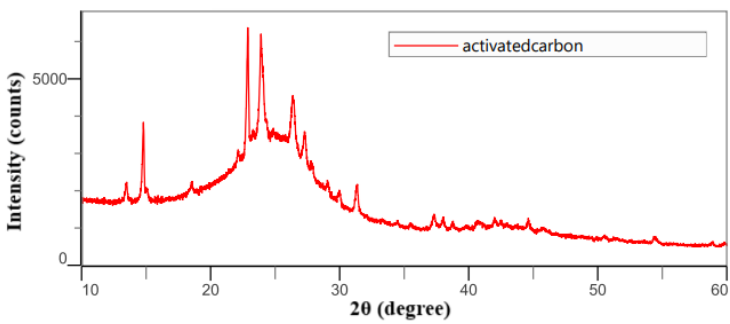
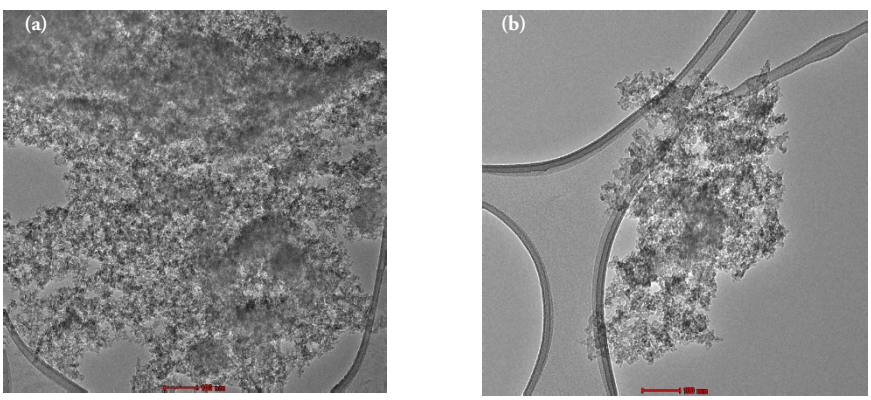


Figure 3: XRD diffractogram pattern of *Areca catechu* husk AC.

Analysis of the XRD pattern in Figure 3 revealed two broad peaks centred at 2θ = 26° and 43°, corresponding to (002) and (101) lattice planes of graphitic structures, which are characteristic features of ACs.[26] Enhanced background scattering between 2θ = 10°–20° suggests the presence of micropores, while sharp peaks at 23° and 27° indicate residual crystalline quartz from the carbonisation process in the furnace.[27] The peak at 24° can be attributed to silicon diphosphate species formed during phosphoric acid activation.[28,29] The diffraction pattern showed elevated background intensity in the 2θ range of 10°–20°, indicating extensive micropore development within the carbon structure.[30] This background enhancement results from X-ray scattering by micropore walls, providing direct evidence of the material's porosity.[31]

The broad nature of these diffraction peaks indicates the material's predominantly amorphous character and limited graphitisation, which is typical for ACs designed for adsorption applications.[32] This structural characteristic aligns with our moderate carbonisation temperature (500°C), which promotes partial graphitisation while preserving the pore network.[33] The amorphous nature enhances the material's practical utility, as such structures generally demonstrate enhanced adsorption performance due to their increased surface area and higher concentration of surface-active sites.[34] These observations demonstrate how the chemical activation process facilitates pore structure development, which is essential for the material's pollutant adsorption capabilities.

3.3 Electron Microscopy: SEM and TEM Observations



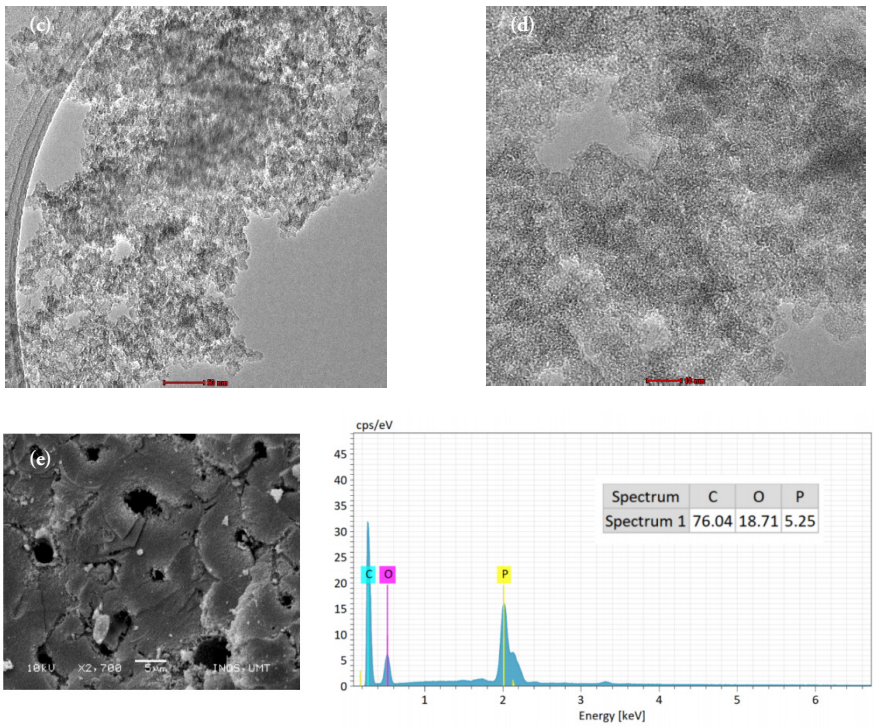


Figure 4: SEM images of the prepared AC at different magnifications: (a–b) 100 nm; (c) 50 nm; (d) 10 nm; (e) EDX analysis.

SEM images (see Figure 4a–4d) revealed irregular pores and cavities on the AC surface, ranging from 1 μm–4 μm in diameter, indicating the presence of hierarchical porous structures with both mesopores and micropores. EDX analysis (see Figure 4e) showed the presence of common elements found in biomass AC, namely carbon (C), oxygen (O) and phosphorus (P). This elemental composition aligns with previous studies on biomass-derived ACs, though specific ratios vary based on precursor material, activation agent and the specific methodology.[35] Biomass-derived ACs are well-established to generally comprise of C, O, nitrogen (N), P and silicon (Si) as the main elements present.[36] Some elements may be in trace quantities and thus remain undetected during analysis due to the low signal intensity, which helps explain any apparent absences in the results.

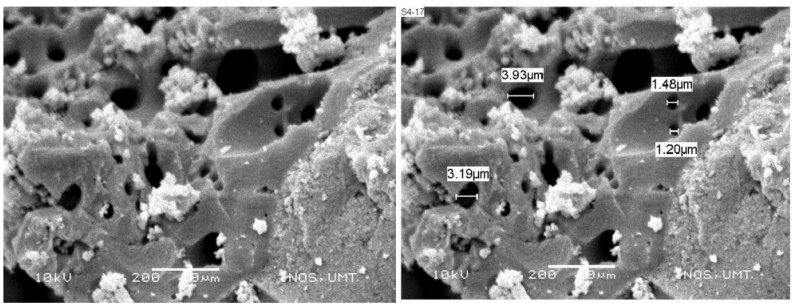


Figure 5: TEM of the AC. From high-resolution TEM (see Figure 5) image, the prepared AC featured randomly oriented graphitic-like carbon layers with an interlayer spacing of 0.294 nm. Given that AFs molecules measure approximately 1.08 nm × 0.87 nm, the pore structure appears well-suited for AFs capture, potentially allowing for high specificity in removing AFs from aqueous solution.[37,38]

3.4 Surface Textural Properties: N Adsorption/Desorption

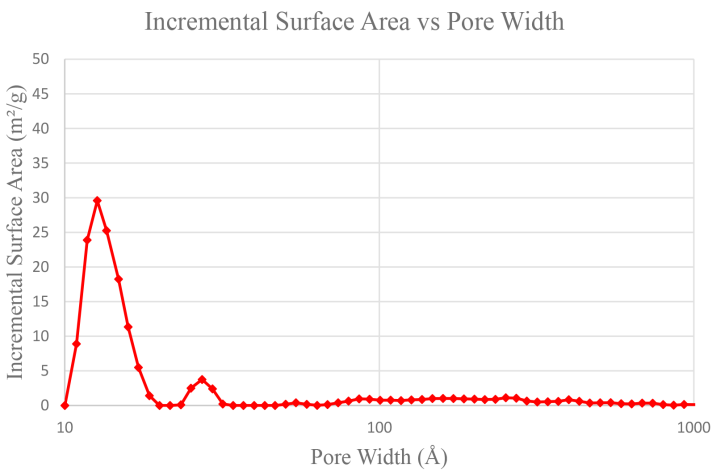


Figure 6: Pore size distribution curve of the *Areca catechu* husk AC.

Figure 6 illustrates the pore size distribution curve of the *Areca catechu* husk AC by using the BET method. From the curve, it is shown that micropores with pore size below 2 nm are predominant, while a significant concentration of micropores around 0.5 nm is also present. Some macropores with pore size larger than 100 nm can also be observed. From the pore size distribution curve, it can be concluded that the prepared AC exhibits a highly porous nature with varied pore size that features a large amount of micropores, followed by mesopores and a low concentration of macropores.

Table 1: The surface properties of the synthesised AC

Activated carbon	Surface Area (m^2/g) (SBET)	Surface Area (m^2/g) (SBJH)	% (SBJH/ SBET)	Pore volume (cm^3/g) (VBJH)	Total pore volume (cm^3/g) VT (0.95)	% (VBJH/ VT)	Reference
Banana peels	289.86	113.348	39.1016	0.21817	0.249	87.5502	37
Pomegranate peels	183.89	125.113	68.0352	0.42801	0.462	92.6407	37
Areca catechu husk	699.53	293.610	41.9700	0.81000	1.03000	78.6400	Our work

Note: SBET= BET specific surface area; SBJH = BJH surface area ; VBJH= BJH pore volume ; VT = total pore volume.

BET and BJH techniques were employed to study the nitrogen gas adsorption and desorption on the AC (see Table 1). *Areca catechu* husk AC prepared in this study exhibited a maximum surface area (SBET) of 699.53 m^2/g, maximum total VT of 1.03 cm^3/g and SBJH of 293.61 m^2/g representing 41.97% of the total surface area. When compared to other ACs, the current AC demonstrated a superior removal capability for AF. From Table 1, we can observe that different biomass sources yield unique ACs which exhibit distinct surface and textural properties.[37] Individual differences in the initial biomass composition serve as excellent precursors for generating ACs with diverse types and functionalities, aligning with our current findings.[39]

3.5 Optimisation of Removal Time

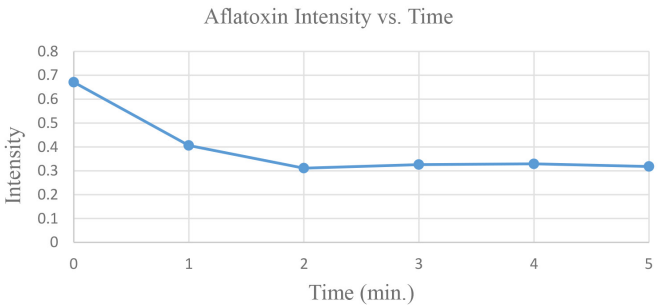


Figure 7: Graph of AFs fluorescence intensity against time, showing a complete removal within 2 min.

The relationship between AFs fluorescence intensity and adsorption duration is as shown in Figure 7. The data demonstrates that our synthesised AC completely removed 60 ppb AFB_1 beneath the limit of quantification within a brief 2 min period. This performance shows substantial improvement over alternative detoxification methods discussed earlier, where UV or visible light degradation processes typically require around 30 min, while chemical or enzymatic treatments can extend to several hours.

When considering that AFs are detected in surface waters, treated water and industrial wastewater up at concentrations up to 1.8 ppb, the efficiency of our AC becomes significant.[10] The material's ability to completely remove 60 ppb of AFB_1 indicates its potential effectiveness in treating real-world contamination levels, which are typically much lower. This suggests that the AC could achieve complete or near-complete removal of AFs from contaminated water sources under practical conditions, making it a viable solution for water treatment applications.

Additionally, the AC developed in this study offers a notable advantage as it does not require pH adjustment to optimise removal efficiency or kinetics. This characteristic proves particularly valuable for practical applications, especially in industrial wastewater treatment, where minimising additional processing steps is crucial for operational efficiency and cost-effectiveness.

4. CONCLUSION

AC derived from *Areca catechu* husk has demonstrated efficient removal of AFB_1 (60 ppb) from water and exhibits several advantages over other adsorbents in AFs removal. These benefits include high adsorption capability, inexpensive and widely available raw material sourcing, simple storage requirements, and most significantly, effectiveness without pH adjustment, which is a crucial factor for wastewater applications. Furthermore, our *Areca catechu* husk-derived AC showed exceptional efficiency in AFB_1 remediation, achieving complete removal within 2 min, surpassing the performance of other AFs degradation or adsorption methods previously reported in literature.[8,9] This exceptional performance aligns with the growing body of evidence demonstrating that AC derived from agricultural waste such as nipa palm and rubber seed shells can effectively remove various organic pollutants from aqueous solutions.[40,41] We anticipate that this strategy of developing adsorbents from agricultural waste shows high promise for targeting and removing other hazardous contaminants in industrial wastewater. Future research directions should focus on improving AC through optimisation of parameters such as impregnation and time, aiming to better understand the mechanisms of AFs adsorption.

5. ACKNOWLEDGEMENTS

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