Parthenium hysterophorus-Derived Nano-Filter for Pharmaceutical Contaminant Removal

A preliminary study using ciprofloxacin

Abstract

Pharmaceutical residues in water create a need for treatment materials that can make use of unwanted plant biomass. This study evaluated carbonized *Parthenium hysterophorus* as a filter-paper coating, using ciprofloxacin as the test pharmaceutical. The objective was to determine whether the coated filter could produce a measurable change in the UV-visible signal during flow-through filtration. Carbon was prepared using a choline chloride-ferric chloride deep eutectic solvent and solvothermal treatment at 200°C for 18 hours. Approximately 15 mg of carbon was dispersed in ethanol, sonicated and deposited onto Whatman Grade 41 paper by vacuum filtration. A 25 mL ciprofloxacin feed at a nominal concentration of 100 mg/L passed through the coated paper in 9 minutes 30 seconds. Initial and filtrate samples were analysed by UV-visible spectroscopy after identical twofold dilution. At 272 nm, absorbance decreased from 2.190 to 1.368, corresponding to a 37.5% relative absorbance decrease. Lower absorbance was also observed across the neighbouring principal-band wavelengths and at 320 nm. FTIR was used to characterize the prepared carbon, with band regions consistent with O-H, aliphatic C-H and C-O vibrations. These preliminary findings provide a measurable optical result for the coated-filter system and a basis for concentration optimisation and controlled evaluation of pharmaceutical contaminant treatment.

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1 Introduction

Pharmaceutical contamination is a growing concern for water quality. Active pharmaceutical ingredients enter waterways through the manufacture, use and disposal of medicines. A global survey published in 2022 analysed 61 pharmaceuticals at 1,052 sites along 258 rivers in 104 countries. At 25.7% of the sampled sites, at least one compound exceeded an aquatic-effect or antimicrobial-resistance concern threshold [2]. These findings show why pharmaceutical residues deserve attention even when water appears clear.

Parthenium hysterophorus was selected as the plant precursor because this problematic weed can provide biomass for a carbon-based material. The untreated plant is associated with allergic contact dermatitis [3]. Converting its biomass into a filter coating connects the use of an unwanted plant with the treatment of pharmaceutical-contaminated water. In this study, carbon was prepared, deposited on paper and tested with a ciprofloxacin feed passing through the coating.

The experiment used one Ciprodac 500 tablet labelled Ciprofloxacin Tablets IP 500 mg, crushed and made up to 100 mL with water to prepare a nominal 5000 mg/L stock based on the labelled content. A 0.5 mL aliquot was mixed with 24.5 mL deionised water to prepare the 25 mL feed. Approximately 15 mg of prepared carbon was dispersed in 20 mL ethanol and sonicated in two 15-minute cycles before deposition onto Whatman Grade 41 paper. The carbon preparation included washing and drying after solvothermal treatment. The complete filtration run lasted approximately 570 seconds. Initial and filtrate samples each received 1 mL sample plus 1 mL water before measurement on a Shimadzu UV-1900 spectrophotometer. This connects carbon preparation, paper coating and measurement of the liquid before and after filtration in a defined workflow.

The broader aim is pharmaceutical contaminant treatment, with ciprofloxacin providing the initial screening result. This preliminary test provides a documented preparation and filtration workflow for further evaluation. Tests involving Benadon, Citzen and other pharmaceutical preparations are underway, and their results are pending. The next stage will compare concentrations and coating amounts while extending the evaluation to additional compounds.

2 Study objective

The objective was to prepare and test a carbonized *Parthenium hysterophorus* coating on filter paper for pharmaceutical contaminant treatment. Ciprofloxacin was used to test whether passing its aqueous tablet preparation through the coated paper would produce a measurable decrease in the UV-visible signal.

Three parts of the experiment were documented: preparation of the plant-derived carbon, deposition of the carbon onto the paper support, and comparison of the initial and filtered samples. The matched-wavelength absorbance decrease at 272 nm was used as the main numerical result, because the initial principal band reached its maximum there. Nearby wavelengths and 320 nm were also examined to describe the spectral change. The working hypothesis was that the coated paper would reduce the measured signal. Adsorption onto carbon was considered a possible explanation to investigate through controlled follow-up tests.

3 Materials and methods

3.1 Materials and experimental scope

Parthenium hysterophorus leaves were collected locally. The materials included leaf powder, choline chloride, ferric chloride, hydrochloric acid, distilled or deionised water, ethanol, potassium bromide for FTIR sample preparation, and Ciprodac 500 tablets labelled Ciprofloxacin Tablets IP 500 mg. Whatman Grade 41 ashless

filter paper was used as the coating support. Its product package identified catalogue number 1441-125 and a supplied circle diameter of 125 mm.

Preparation, filtration and measurements were carried out using a mortar and pestle, a heated preparation stage, a Teflon-lined stainless-steel autoclave, an oven, a sonicator, a vacuum filtration arrangement, a hydraulic press for FTIR pellet preparation and a Shimadzu UV-1900 spectrophotometer. FTIR was also used to characterize the carbon after preparation.

Table 1 summarises the quantities and operating conditions used. It distinguishes the tablet stock, the filtration feed and the diluted initial sample used for UV-visible measurement.

Table 1 Recorded preparation and filtration conditions

Parameter	Recorded value
Leaf powder used for synthesis	2 g
Deep eutectic solvent	4 g; choline chloride-FeCl3 at 1:2 molar ratio
Initial heating	80°C for 1 hour
Solvothermal treatment	200°C for 18 hours
Final carbon drying	80°C overnight
Carbon introduced for coating	Approximately 15 mg in 20 mL ethanol
Sonication	Two 15-minute cycles
Support	Whatman Grade 41 paper; catalogue 1441-125
Nominal stock concentration	5000 mg/L
Nominal feed concentration and volume	100 mg/L; 25 mL
Total filtration run time	9 minutes 30 seconds
Dilution before UV-visible measurement	1 mL sample plus 1 mL water for both samples

3.2 Preparation of solvothermal carbon

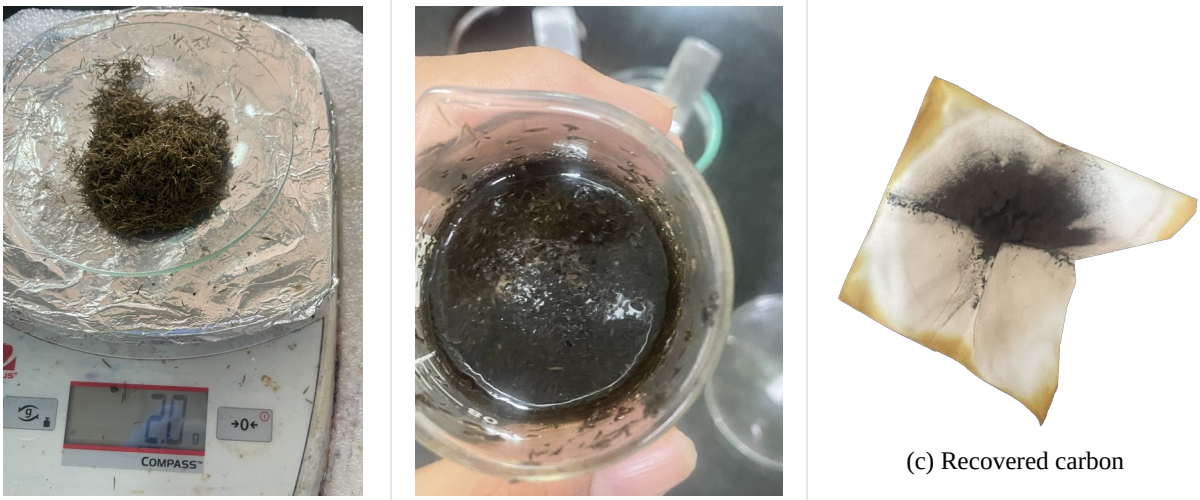
The carbon was prepared using the published solvothermal procedure [1]. *Parthenium hysterophorus* leaf powder (2 g) was combined with 4 g of a choline chloride-FeCl3 deep eutectic solvent prepared at a 1:2 molar ratio. The mixture was heated at 80°C for 1 hour to obtain a semi-solid material, then added to 50 mL distilled water. The mixture was transferred into a sealed Teflon-lined stainless-steel autoclave and treated at 200°C for 18 hours.

The 1:2 ratio describes the molar proportions of choline chloride and ferric chloride. The 4 g quantity refers to the combined solvent mixture, not 4 g of each ingredient. The 2 g quantity describes the leaf powder introduced at the start of synthesis.

The initial heating and sealed-vessel treatment were carried out as separate stages. The plant powder and solvent were first heated at 80°C, then the semi-solid mixture was added to the 50 mL of distilled water before autoclave treatment. After the 18-hour treatment, the vessel was allowed to cool to room temperature.

The dark-brown solid was recovered by filtration through Whatman filter paper. The recovered material was washed three times with 1 M hydrochloric acid, three times with distilled water and twice with an ethanol-water mixture at a 4:6 ratio. It was then dried at 80°C overnight to obtain a fine carbon powder.

The washing sequence was hydrochloric acid, water and then the ethanol-water mixture. The washed and oven-dried solvothermal carbon was used to prepare the coating dispersion. Figure 1 shows the weighed plant material, the reaction mixture and the recovered solid.



(a) Plant material weighing (b) Reaction mixture (c) Recovered carbon

Figure 1 Preparation of carbonized *Parthenium hysterophorus*. (a) Weighing of 2.0 g plant material. (b) Reaction mixture containing *Parthenium hysterophorus*, choline chloride and ferric chloride with 50 mL distilled water. (c) Solid recovered on filter paper after the autoclave treatment.

3.3 Preparation of ciprofloxacin stock and feed

One Ciprodac 500 tablet labelled Ciprofloxacin Tablets IP 500 mg was crushed into a fine powder using a mortar and pestle. The powder was combined with water, and the preparation was made up to a final volume of 100 mL. From the labelled drug content, the nominal stock concentration was calculated as 500 mg divided by 0.100 L, giving 5000 mg/L.

The stock concentration was calculated based on the labelled ciprofloxacin content. The final preparation volume was 100 mL. The resulting nominal stock concentration was 5 mg/mL, equivalent to 5000 mg/L.

A 0.5 mL stock aliquot was transferred and mixed with 24.5 mL deionised water to prepare 25 mL of feed. This 50-fold dilution gave a nominal concentration of 100 mg/L, approximately 100 ppm in a dilute water sample. Nominal concentration refers to the concentration calculated from the tablet label and preparation volumes.

The dilution calculation was checked from the amount transferred. At 5 mg/mL, the 0.5 mL stock aliquot corresponded to 2.5 mg ciprofloxacin. Dividing 2.5 mg by the final 25 mL gave 0.1 mg/mL, or 100 mg/L. This 25 mL feed was used for the filtration test.

3.4 Dispersion and deposition of the carbon coating

Approximately 15 mg of the carbon powder was weighed and combined with 20 mL ethanol. This gave an introduced dispersion concentration of approximately 0.75 mg/mL. The dispersion was sonicated in two 15-minute cycles, giving 30 minutes of sonication in total.

Ethanol was used to disperse the carbon before deposition. The sonicated mixture was passed through the mounted Whatman paper using a vacuum pump. As the liquid passed through, carbon accumulated on the paper to form the coating. Figure 2 shows the sonication stage, the coating assembly and the coated paper supports.

The coated paper was then used for the aqueous ciprofloxacin feed. The approximately 15 mg quantity refers to the carbon powder weighed into the dispersion before deposition onto the paper.

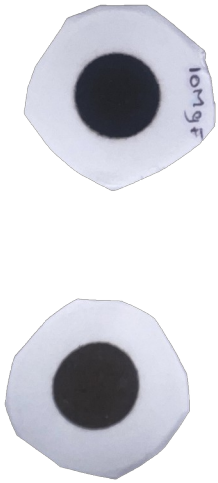
Whatman Grade 41 paper identified by catalogue number 1441-125 was used. The supplied paper-circle diameter was 125 mm. This value describes the product dimension rather than the area exposed to flow in the mounted filtration assembly.



(a) Sonication



(b) Vacuum coating assembly



(c) Coated filter papers

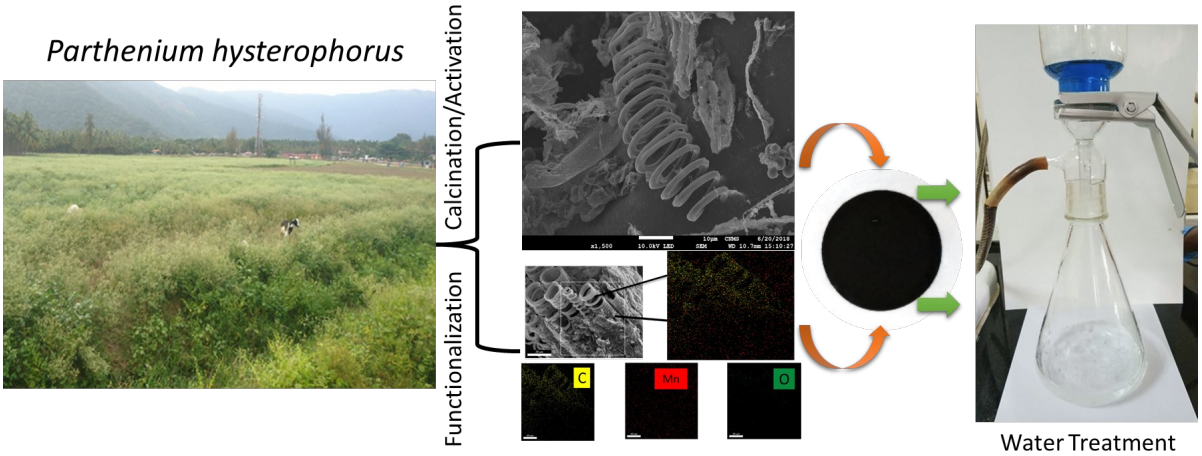


Figure 2 Preparation and deposition of the carbon coating. (a) Sonication stage for the dispersion prepared with approximately 15 mg carbon in 20 mL ethanol, using two 15-minute cycles. (b) Continuous-flow coating assembly connected to a vacuum pump. (c) Appearance of coated paper supports. The stated carbon mass is the powder weighed into the coating dispersion.

3.5 Filtration of the feed

The entire 25 mL ciprofloxacin feed was passed through the coated paper under vacuum. The complete run took approximately 9 minutes 30 seconds, or 570 seconds. From 25 mL divided by 9.5 minutes, an approximate average flow rate of 2.63 mL/min was calculated.

The total run time was used to calculate this whole-run average. The 570-second value describes the passage of the full feed through the assembly; it is not a measurement of the time an individual molecule spent in contact with the carbon.

The filtrate was collected and sampled for the post-filtration UV-visible comparison. Its spectrum was compared with the initial feed spectrum after applying the same dilution to both samples.

3.6 UV visible measurements

Each UV-visible measurement sample was prepared by combining 1 mL of sample with 1 mL water. This dilution was used to reduce absorbance and improve the clarity and interpretation of the spectral peaks. This twofold dilution was applied to both the initial feed and the collected filtrate before measurement on a Shimadzu UV-1900 spectrophotometer. The nominal concentration of the diluted initial sample in the cuvette was 50 mg/L.

Paired initial and filtrate spectra were collected from 200 to 800 nm at a wavelength spacing of 2 nm. Identical 1:1 sample-to-water dilution was used for both traces so that absorbance could be compared directly at the same wavelengths.

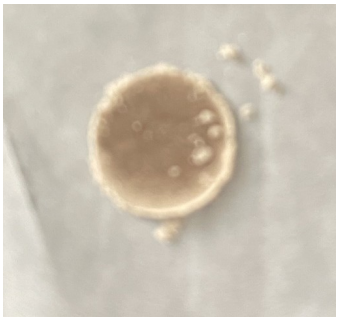
The 200-400 nm interval was plotted, where the main measured bands were visible. The recorded data points were joined directly, without smoothing, baseline subtraction or separate scaling of the two traces. Figure 4 shows the paired spectra.

3.7 FTIR characterization

FTIR was performed on *Parthenium hysterophorus* after solvothermal carbonization, washing and drying. The carbon was mixed with potassium bromide (KBr), and the mixture was compressed into a pellet using a hydraulic press. Figure 3 shows the press and the prepared pellet. Figure 5 presents the FTIR spectrum of the prepared carbon.



(a) Hydraulic press



(b) Carbon and KBr pellet

Figure 3 Sample preparation for FTIR. (a) Hydraulic press used for pellet preparation. (b) Pellet prepared from the carbon sample mixed with potassium bromide.

3.8 Calculation of the optical change

The initial and filtrate absorbance were compared at the same wavelength. The initial principal band reached its highest sampled absorbance at 272 nm. The percentage decrease was calculated by subtracting the filtrate absorbance from the initial absorbance, dividing by the initial absorbance and multiplying by 100.

Matched wavelengths were used for every percentage calculation. The final trace's own maximum was recorded separately rather than substituted into the 272 nm calculation. The percentage describes the relative optical change between the two traces. It was used as a screening result rather than a calibrated concentration-removal measurement.

4 Results

4.1 Change in the measured spectrum

Lower filtrate absorbance was observed around the principal ultraviolet band. At 272 nm, the initial absorbance was 2.190 and the filtrate absorbance was 1.368. The absolute decrease was 0.822. The relative absorbance decrease was 37.53%, reported as 37.5%.

$$Absorbance\ \ decrease\ (\%) = \frac{2.190 - 1.368}{2.190} \times 100 = 37.53\%$$

The initial principal-band maximum occurred at 272 nm. The filtrate principal-band maximum occurred at 270 nm, with absorbance 1.384. This was a difference of one wavelength sampling interval. Matched wavelengths were used for the numerical comparisons in Table 2.

Table 2 Matched wavelength absorbance readings and descriptive decreases

Wavelength nm	Initial absorbance	Filtrate absorbance	Absorbance decrease %
270	2.133	1.384	35.1
272	2.190	1.368	37.5
274	2.144	1.317	38.6
320	0.810	0.535	34.0

A decrease was also observed at 320 nm: absorbance changed from 0.810 to 0.535, corresponding to 34.0%. The magnitude of the optical decrease therefore differed across the spectrum.

At 270 nm, absorbance decreased from 2.133 to 1.384, or 35.1%. At 274 nm, it decreased from 2.144 to 1.317, or 38.6%. Together with the 272 nm result, these values show a decrease across the sampled main band rather than at only one selected wavelength.

At 272 nm, the filtrate absorbance was approximately 62.5% of the initial absorbance. This is the complement of the 37.5% optical decrease. The wavelength-specific values were retained rather than combined into one spectrum-wide removal percentage.

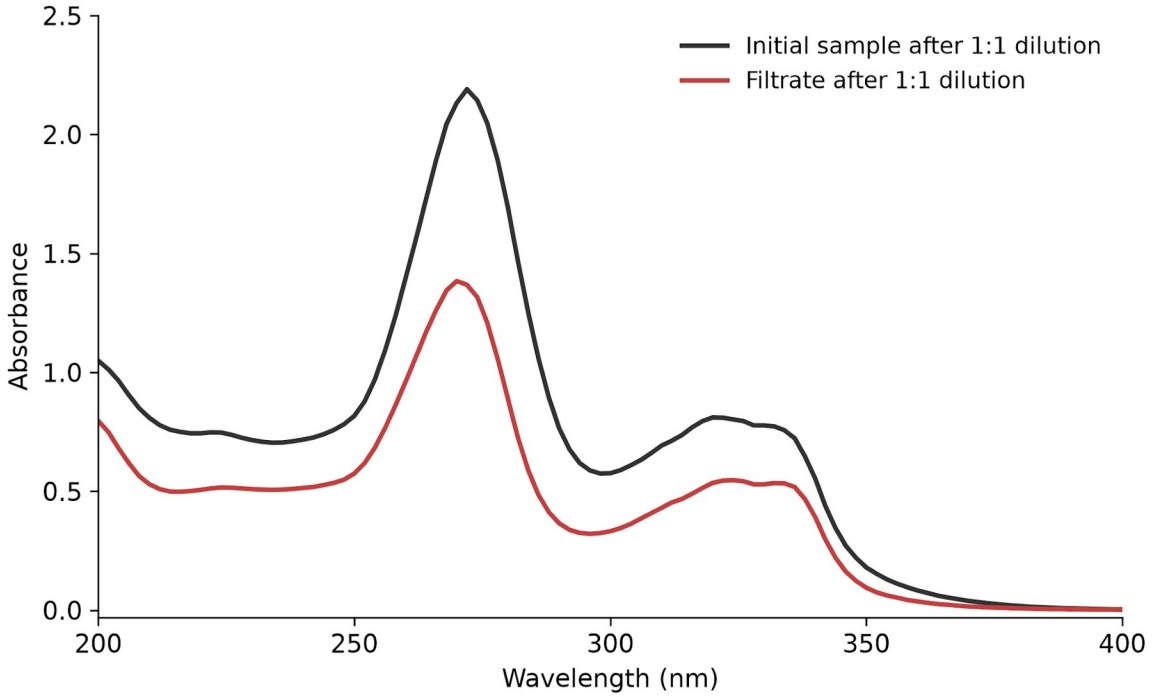


Figure 4 Initial and filtrate UV-visible spectra after identical 1:1 dilution. The plotted interval is 200-400 nm from the 200-800 nm scan. Recorded data points are joined directly without smoothing, rescaling or baseline correction.

4.2 FTIR characterization

The FTIR spectrum of the carbonized *Parthenium hysterophorus* sample was examined after solvothermal preparation, washing and drying. The displayed wavenumber axis extends approximately from 4000 to 500 cm-1. The plotted values on the axis labelled percentage transmittance exceed 100. The visible band positions were therefore used for tentative interpretation rather than treating the vertical values as validated absolute percentage transmittance.

The observed band regions were assigned to the following characteristic vibrations; overlapping contributions are considered in the interpretation.

Table 3 FTIR band regions and assignments

Wavenumber region (cm-1)	Band assignment
3400-3450	O-H stretching region
2920	Aliphatic C-H stretching
1640	Aromatic or conjugated vibrations and water-bending region
1400	C-H bending region
1100-1000	C-O and C-O-C stretching region

The FTIR band assignments provide a qualitative description of the chemical features of the prepared carbon.

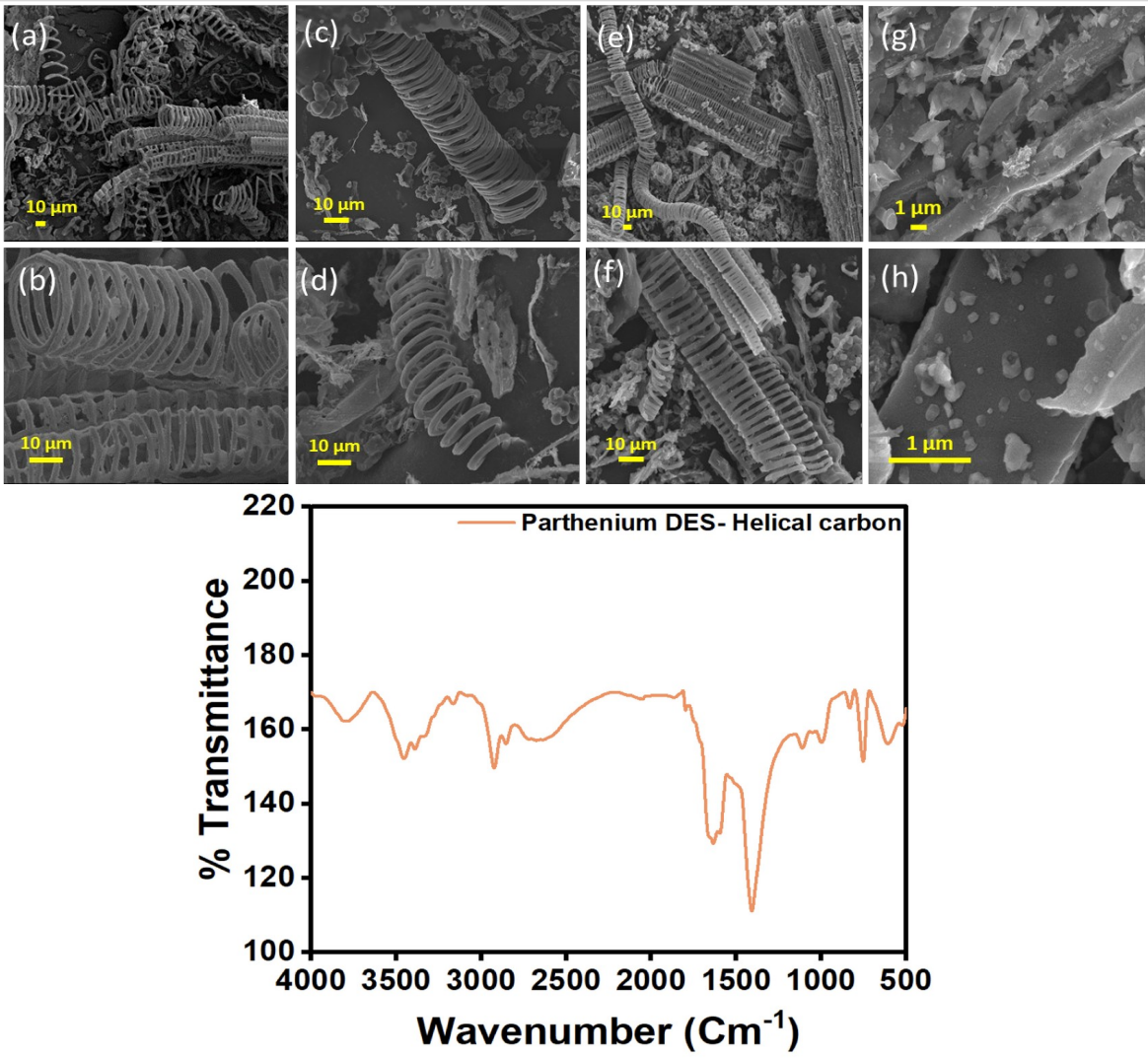


Figure 5 FTIR spectrum of *Parthenium hysterophorus* after solvothermal carbonization, washing and drying. The carbon-KBr pellet was prepared for the measurement. The spectrum is reproduced with its original axes and signal.

5 Discussion

The main result was a decrease in UV-visible absorbance after passage of the ciprofloxacin feed through the carbon-coated paper. At 272 nm, the relative decrease was 37.5%, with lower readings at neighbouring wavelengths and at 320 nm. The same dilution was used for both samples, and readings were compared at matched wavelengths. This provides an initial optical result for the next tests of the filter system.

Adsorption onto carbon is a possible explanation for the decrease. FTIR was used to describe possible chemical features of the prepared carbon. Controlled comparisons will be used to examine the contributions of the carbon, paper support and sample matrix.

A connected workflow was developed from the plant precursor to the coated paper and the filtration test. Preparation quantities, heating stages, washing sequence, coating dispersion, sonication time, feed dilution and

total filtration duration were recorded. Separating the 100 mg/L feed from the 50 mg/L initial cuvette sample makes the concentration calculations clear for repeating the experiment.

Coated and uncoated paper will be compared using the same feed and filtration arrangement. Water will also be passed through coated paper to examine any material released by the coating. Independent repeats and a concentration calibration will help determine reproducibility and quantify the effect added by the carbon coating.

The pharmaceutical-removal study will be extended compound by compound, keeping the coating preparation and measurement dilution consistent. For each pharmaceutical, the preparation and concentration will be specified, and an analytical method suited to that compound will be used. This will allow comparison of outcomes without assigning the ciprofloxacin result to other medicines.

Tests involving Benadon, Citzen and other pharmaceutical preparations are underway, and their results are pending. These tests are not included in the current results.

6 Future Scope and Immediate Next Steps

The 37.5% relative absorbance decrease was a preliminary result from the initial ciprofloxacin test. Follow-up experiments will vary the pharmaceutical feed concentration and carbon coating amount to identify conditions that give the strongest performance. Tests involving Benadon, Citzen and other pharmaceutical preparations are underway.

Independent repeats, coated and uncoated paper comparisons, a coated-paper water blank and concentration calibration will be carried out. Retained carbon mass and filtrate recovery will be measured, and the FTIR vertical scaling will be checked to strengthen quantitative analysis.

7 Conclusion

Carbonized *Parthenium hysterophorus* was prepared through solvothermal treatment and deposited on Whatman Grade 41 paper. A 25 mL feed of nominal 100 mg/L ciprofloxacin was passed through the coated paper in approximately 9 minutes 30 seconds. With identical measurement dilution, absorbance at 272 nm decreased from 2.190 to 1.368, giving a relative absorbance decrease of 37.5%. Lower absorbance was also measured at the other selected wavelengths.

This initial result supports further testing of the coating for pharmaceutical contaminant treatment. A preparation and flow-through testing procedure has been established for repeated experiments and comparison with controls. The next goal is to quantify the carbon coating's contribution using repeated experiments and a validated measurement of drug concentration.

8 Data availability and contribution

The paired spectra were used to calculate the matched-wavelength absorbance changes and to prepare the UV-visible graph.

Photographs were cropped and background-isolated for presentation; original sample detail was retained.

Material preparation, coating, filtration and measurements were carried out under laboratory supervision.

9 References

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