Introducing a low-cost jute activated carbon as a novel cleanup agent in multiclass pesticide residue analysis using gas chromatography tandem mass spectrometry

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ABSTRACT

For the first time, jute activated carbon (JAC) derived from jute stick, a byproduct of the jute industry, was characterised and evaluated for its cleanup efficiency in a mixture of 181 multiclass pesticide residue testing using gas chromatography tandem mass spectrometry (GC-MS/MS). Four commercially-important food matrices including okra, spinach, pomegranate and tea were chosen. Various physico-chemical techniques were used to characterise the material. The cleanup method involved dispersive solid phase extraction (dSPE) using a combination of JAC (5 mg) with 25 mg of primary secondary amine per mL of the sample extract (in ethyl acetate). The findings demonstrated a lower matrix effect and higher signal-to-noise ratio were recorded for JAC. Overall, the method offered satisfactory recoveries for most of the pesticides in all the tested matrices. The cleanup effectiveness of JAC showed superiority over the commercially available, non-renewable dSPE sorbent *viz.* graphitised carbon black (GCB). With a production cost of only US Dollar ~10/kg, JAC is a low-cost alternative to commercial GCB (cost = US Dollar 11–12/g). The study valorises the potential of JAC and anticipates its large-scale application in food testing laboratories.

1. Introduction

Agricultural byproducts generate a huge amount of waste which, in most cases, pose a serious economic and ecological issue; such an agricultural by-product is jute stick which is generated from the 'golden fibre' of jute (*Corchorus* sp.). As the second most available natural fibre in the world after cotton, jute is largely cultivated in Asian countries including India, Bangladesh, Pakistan and China. Jute fibre is extracted from the harvested jute plants (90–120 days after sowing) by the retting process. Owing to its high tensile properties and biodegradability, jute fibre is well known since ages for its packaging applications including sacking and hessian. Furthermore, its recent applications in bio-composite, paper, geotextiles, agro-textiles and life-style products have created new market opportunities for jute fibre (Ray and Ghosh, 2018).

Because the lignocellulosic biomass of jute stick is neither suitable as a fodder nor for manure preparation, farmers always face hurdles to store such a voluminous agro-residue either on-site or off-farm (Ray and Ghosh, 2018). In practice, the biomass of jute stick is either dumped or burned, causing environmental pollution in nearby localities. Currently, India, one of the largest producers of jute, generates approximately 4 MT of jute stick every year (Ghosh et al., 2019). With a very limited commercial significance and its menace, finding some way out to manage this agro-waste would be of great utility.

Activated carbon (AC) obtained from various carbonaceous sources possesses high porosity, and for this, it has been widely utilised as an effective adsorbent for the removal of pollutants predominantly from water bodies and air (Vinod and Imran, 2000; Paul Chen et al., 2003). Usually, the commercial ACs are sourced from various raw carbon

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resources (*e.g.* coal, peat and lignite) as well as biomass resources (*e.g.* coconut shells and wood) (Ioannidou and Zabaniotou, 2006; Hayashi et al., 2000). Among various ACs, the petroleum-derived graphitised carbon black (GCB) (Narenderan et al., 2020; Samsidar et al., 2018) is most commonly used in pesticide residue analysis. As it is highly priced and non-renewable in nature, there is a growing interest in the production of low-cost ACs from renewable and cheaper precursors from agricultural wastes including rice husks (Sahu et al., 2009), date pits (Bouchelta et al., 2012), groundnut shell (Malik et al., 2006), bamboo (Hameed et al., 2007) and oil palm fibre (Tan et al., 2007), just to name some. Considering their large surface areas, these ACs have mostly been utilised in waste water treatment. In comparison, jute stick (predominantly a lignocellulosic material) has received very little attention for AC production (Ghosh et al., 2020). In order to make a better use of this abundant and underutilised agro-waste (a byproduct of jute industry), we aimed to prepare jute activated carbon (JAC) from jute stick and investigate its potential utilisation as a sorbent material for cleanup in pesticide residue analysis, and as a low-cost alternative to GCB.

In recent years, the contamination of foods by pesticide residues causes serious environmental menace due to their frequent applications in crops and fields. Given the toxicity of pesticides, various regulatory bodies have fixed their maximum residue limits (MRLs) in food commodities across the nations, demanding effective multiresidue analytical methods for the surveillance and testing of samples for MRL compliance. For any residue analysis method, accuracy and precision are the two major performance parameters. One of the serious problems that the residue chemists routinely face is the matrix effect, resulting in inaccurate estimation of pesticide concentrations. A cleanup step is, therefore, necessary to remove the co-extracted, matrix-derived compounds and minimise their influence on the extent of signal enhancements or suppressions of target pesticides. Over the past decade, the analysis of pesticide residues has mostly involved the QuEChERS and ethyl acetate extraction-based sample preparation methods (Anastassiades et al., 2003; Khan et al., 2014; Lawal et al., 2018), where the cleanup has been performed through the process of dispersive solid phase extraction (dSPE). In dSPE, researchers used adsorptive carbon to remove co-extracted plant pigments that interfered during the instrumental analysis (Islam et al., 2019; Hou et al., 2014). Earlier, some also used activated carbon as an adsorbent in combination with primary secondary amine (PSA) to take out the matrix-derived compounds, especially the co-extracted plant pigments and fatty acids (Li et al., 2009; Lawal et al., 2018).

Elsewhere, researchers also showed GCB's strong affinity for the planer pesticides (Mol et al., 2007; Rejczak and Tuzimski, 2015), resulting in their recovery loss. Considering this limitation, several adsorbent materials such as surface-modified silica (Peng et al., 2010), multi-walled carbon nanotubes (Hou et al., 2014; Zhao et al., 2012), amine-functionalized magnetic nanoparticles and multiwalled carbon nanotubes (Deng et al., 2014), amine-modified graphene (Guan et al., 2013) and magnetic graphene (Luo et al., 2015) were developed and evaluated as the alternatives of GCB. As these materials suffer from some or other disadvantages (*e.g.* high cost and inadequate availability), a further search for an effective cleanup agent continues.

Previously, a group of researchers revealed how phenolic resin-based ACs could be applied as a reversed-dispersive solid phase extraction (r-dSPE) sorbent. They showed that the type of AC determined 26 pesticides in food matrices (*e.g.* cauliflower, cucumber, banana, apple, wheat and black gram) in addition to acting as a cost-effective dSPE reagent (Singh et al., 2018). Elsewhere, researchers reported the effectiveness of jute biomass carbons in the removal of heavy metals and textile dyes (Asadullah et al., 2010, 2014). In our earlier study, we demonstrated that jute stick derived activated carbon (with a surface area greater than 1000 m^2/g) holds immense potential as an efficient and reusable adsorbent to treat Cd(II) contaminated aqueous medium before the contaminant is released in the environment (Ghosh et al., 2020). Based on these studies, we anticipated that JAC might be useful as an effective

dSPE cleanup sorbent for the removal of matrix-derived phytochemicals, particularly which interfere with detection and quantification of pesticides during residue analysis. As the role of JAC in pesticide residue analysis in foods has never been unfolded, it provided us the initial inspiration to conduct this experiment.

First of its kind, the study describes how JAC was synthesised from jute stick and explores the efficiency of JAC as a cleanup agent for the analysis of multiclass pesticides in four commercially important (APEDA, 2019; Tea Board India, 2018) food matrices including okra (*Abelmoschus esculentus*), spinach (*Spinacia oleracea* L.), pomegranate (*Punica granatum* L.) and tea (*Camellia sinensis* L.), using gas chromatography tandem mass spectrometry (GC-MS/MS). In addition to assessing the structural and chemical characterisation of JAC, its cleanup performance was comparatively evaluated against GCB. This study valorises JAC as a cheap, abundant and efficient alternative to GCB and other r-dSPE sorbents in pesticide residue analysis.

2. Materials and methods

2.1. Preparation of JAC

The raw jute stick used for preparing activated carbon was bought from a local market in Kolkata, India (22.90 N 88.39 E). First, the precursor was cleansed with water and dried in a hot air oven at 105 °C for 24 h. Later, it was crushed into desired mesh size (10 mesh) and stored in a sealed container. Prior to activation, 500 g of the sample was pretreated with 85% ortho-phosphoric acid in the material-to-solution ratio of 10:1. Thereafter, the mixture was heated at 80 °C for 6 h and kept standing overnight at room temperature. Using a muffle furnace (Prime Instruments, Kolkata, India), the treated material was carbonised at 550 °C for 2 h (hold time) under purified nitrogen with a constant heating rate of 5 °C/min. Afterwards, it was brought to the room temperature (28 ± 1 °C) and washed with hot water to take off the activating chemicals until the pH of the washing solution reached 6–7. Later, it was dried in a hot air oven at 105 °C for 24 h, sieved (80 mesh) and stored in a sealed container for conducting further experiments.

2.2. Characterisation of JAC

The lignocellulosic biomass of jute stick was analysed for its various physico-chemical properties including cellulose (TAPPI method- 17), pentosan (TAPPI method 223), uronic anhydride (TAPPI method 209), acetyl content (TAPPI method 209), lignin (TAPPI method 222) fat and wax (TAPPI method 204), ash content (TAPPI method 211), moisture content (TAPPI method 258) and bulk density (TAPPI method 258) with established procedures (TAPPI test methods, 1991). The newly developed JAC was characterised for carbon (%) (CHNS Analyser, Model-Euro EA3000, Italy), ash content (TAPPI method 211), moisture content (TAPPI method 258), bulk density (TAPPI method 258), pH (pH meter, model-FEP20, Mettler-Toledo India Pvt. Ltd, Mumbai, India) and surface area (Smart Sorb 92/93, BET surface area analyser, Smart Instruments Co. Pvt. Ltd., Mumbai, India).

To investigate the nature of functional groups, both the raw jute stick and JAC were analysed by FTIR (Bruker ALPHA FTIR/ATR). At a resolution of 4 cm^{-1} and over the wave-number range of 4000–400 cm^{-1}, the samples were scanned. A total of 24 scans per sample was performed. Using scanning electron microscopy (Evo 18 SEM, Carl Zeiss, Germany) at a working distance of 9 mm with 15 kV electro high tension (EHT), the surface morphology of JAC was characterised. The thermal behaviour of the material was evaluated by thermogravimetric analysis (TGA), where a sample (2.5 mg) was heated (@10 °C/min) under inert (N$_2$) environment on an alumina crucible between 30 °C and 600 °C. The diffraction pattern of JAC was investigated with an X-ray diffractometer (X'pert3 powder, Pananalytical, Netherlands). The XRD analysis was carried out with the Ni filtered Cu anode (λ = 1.5406 Å) at 30 mA current and a voltage of 45 KV within the scan (2θ) range of 5–90.

2.3. Selection of matrices, pesticides and other chemicals

The organically certified food samples (okra, spinach, pomegranate and tea) were obtained from a supermarket in Pune (Maharashtra, India, 18°31′13″N, 73°51′24″E). A total of 181 GC amenable pesticides (from different chemical classes) which are commonly regulated in agricultural crops were chosen. The certified reference standards of each individual pesticide (93–99.99% pure) were purchased from Sigma-Aldrich (St. Louis, MO, USA) and Dr Ehrenstorfer GmbH (Augsburg, Germany). The mass spectrometry-grade solvents (ethyl acetate and water) were procured from Merck (Darmstadt, Germany). The other reagents, for example, acetic acid, anhydrous sodium sulfate and magnesium sulfate were procured from Merck India Ltd. (Bengaluru, India). The dSPE sorbents *viz*. PSA and GCB were obtained from Agilent Technologies (California, USA).

By dissolving 10 ($\pm$0.01) mg of each certified reference standard in 10 mL of ethyl acetate, the primary stock solutions of the pesticides were prepared in a series of certified class 'A' volumetric flasks and then stored in the dark at $-20 \pm 2\,^\circ$C. The intermediate standard mixtures of 1 μg/mL were prepared by appropriate dilution of the primary standards with ethyl acetate and stored in the dark at $-20 \pm 2\,^\circ$C. As per the SANTE guideline (SANTE, 2019), the stability of the standards was intermittently checked. Through appropriately diluting the intermediate standard mixtures with ethyl acetate, the calibration standards (0.01–0.04 μg/mL) were prepared. For the matrix-matched calibration standards, the control matrix extracts of the samples were separately used. With our earlier published ethyl acetate-based extraction method (Khan et al., 2014), the control samples were checked for pesticide content, and no trace of pesticide contamination was found in them.

2.4. Sample preparation

As per our previous protocols (Utture et al., 2012; Banerjee et al., 2012; Khan et al., 2014), the homogeneous matrices were prepared. In brief, the samples (2 kg) were blended in a heavy duty mixer/grinder (Vishvakarma Machine Tools, Rajkot, India). In pomegranate, blending was performed after adding water (1:1, v/v). But in case of tea, the matrix (25 g dry tea) was soaked in 225 mL of water and 0.5% acetic acid for 30 min before comminution. The homogenate (10 g for okra, spinach and pomegranate and 2 g for tea) was extracted using 10 mL of ethyl acetate, which was followed by vortexing (Geni 2T, Vortex mixer, Imperials Biomedicals, Mumbai, India) for 1 min and centrifuging (with a centrifuge from Kubota Corp., Tokyo, Japan) at 2800×*g* for 5 min. To separate the ethyl acetate phase, 10 g anhydrous Na$_2$SO$_4$ was added, vortexed for 1 min and centrifuged at 2800×*g* for 5 min. Thereafter, the extract was subjected to dSPE cleanup with JAC, and lastly, its cleanup performance was evaluated against GCB. For sample preparation, a low-volume concentrator (TurboVap® LV, Caliper Life Science, USA) and micro-centrifuge (Microfuge Pico, Kendro, D-93 37520, Osterode, Germany) were utilised.

2.5. Optimisation and comparison studies for sample cleanup

An aliquot of 1 mL extract spiked at 20 μg/L was drawn and subjected to dSPE cleanup. The performance of JAC and GCB at two rates of 5 and 10 mg/mL was evaluated in combination with PSA (25 mg/mL). In each case, the recoveries were estimated after GC-MS/MS analysis. Besides, a GC-MS full scan analysis was performed to investigate the nature of the co-extracted matrix-derived compounds and the effectiveness of their removal during cleanup.

2.6. Instrumental and analytical conditions

The samples were analysed using a gas chromatograph (GC 2010; Shimadzu Corporation, Kyoto, Japan) hyphenated to a triple quadruple mass spectrometer (TQ8030, Shimadzu Corporation, Kyoto, Japan). For the instrument control, data acquisition and data processing, the GC-MS LabSolutions software (Version 4.11 SU2, Japan) was used. A 1 μL volume of the sample was injected in the splitless mode into an Rxi®-5Sil MS (30 m × 0.25 mm, 0.25-μm film thickness) column from Restek Corporation (Bellefonte, USA) for the chromatographic separation. The ultrapure grade helium was used as the carrier gas at a constant flow rate of 1.2 mL/min, while argon was used as the collision gas. The oven temperature was initially set at 90 °C (1 min), ramped at a rate of 35 °C/min up to 130 °C (8.89 min) and further ramped at the rate of 10 °C/min up to 280 °C (8.89 min), resulting in a total run time of 21.31 min. The transfer line and ion source temperatures were set at 290 °C and 230 °C respectively. The MS/MS data was acquired from 1.5 min onwards. The data acquisition was performed at both scan as well as multiple reaction monitoring (MRM) modes. In the MRM mode, each target pesticide was identified by their specific m/z value that helped further in their selective quantification.

2.7. Matrix effect (ME)

The ME was evaluated (at 20 μg/kg in 6 replicates) by comparing the peak areas of the matrix matched standards (peak area of post-extraction spike) with the corresponding peak areas of solvent standards. Using the following equation, the ME was quantified:

ME (%) = (Peak area of matrix matched standard − peak area of solvent standard) × 100/Peak area of matrix matched standard.

A negative value of ME signifies a matrix induced signal suppression, whereas a positive value signifies an enhancement in signal intensity. The extractable matrix compounds co-eluting with the pesticide molecules were identified on the basis of similarity search with the help of the National Institute of Standards and Technology (NIST) library (version 14) for the mass spectral analysis at their respective retention time (R$_t$).

2.8. Statistical analysis

For every experiment, the values represented the mean of six replicates. The mean values of various treatments were tested by Duncan's multiple range test (DMRT) and using SPSS software, version 16.0. These tests were performed to compare the data related to different doses of adsorbents, matrix-wise recoveries and matrix effects.

3. Results and discussion

3.1. Preparation and characterisation of JAC

The physico-chemical analysis of jute stick showed that it consisted of 40.2% of cellulose, 20.9% of hemicelluloses (including 11.1% of pentosan, 6.2% of uronic anhydrides and 3.6% of acetyl content), 24.1% of lignin, 2.1% of fat and wax, 1.1% of ash and 11.6% of moisture. When the jute stick was processed (see section 2.1), the yield of JAC was 40.8%. Following the CHNS analysis, the carbon enrichment in JAC was 69.2%, although jute stick contained only 45.6% C. Compared to the raw material, the JAC surface turned rougher and cracked with the scanning electron microscopy analysis (Fig. 1a and b). This signifies the presence of well-developed pores in the carbon layers of JAC.

It was previously reported that when lignocellulosic biomass was treated with phosphoric acid (H$_3$PO$_4$), the cellulose, hemicellulose and lignin components of the biomass were degraded, leaving the acid-resistant and cellulosic components of AC as the residual matter (Girgis et al., 2007; Timur et al., 2006). Phosphoric acid degraded the glycosidic linkages of cellulose and hemicelluloses and cleaved the aryl-ether linkages in lignin. This also involved other chemical transformations such as dehydration, depolymerisation, cyclisation and condensation (Timur et al., 2006; Yaket and El-Deen, 2016). The non-carbon components (mainly hydrogen, nitrogen and oxygen) of cellulose, hemicellulose and lignin were converted into various volatile products, which escaped very fast from the surface, leaving the residue
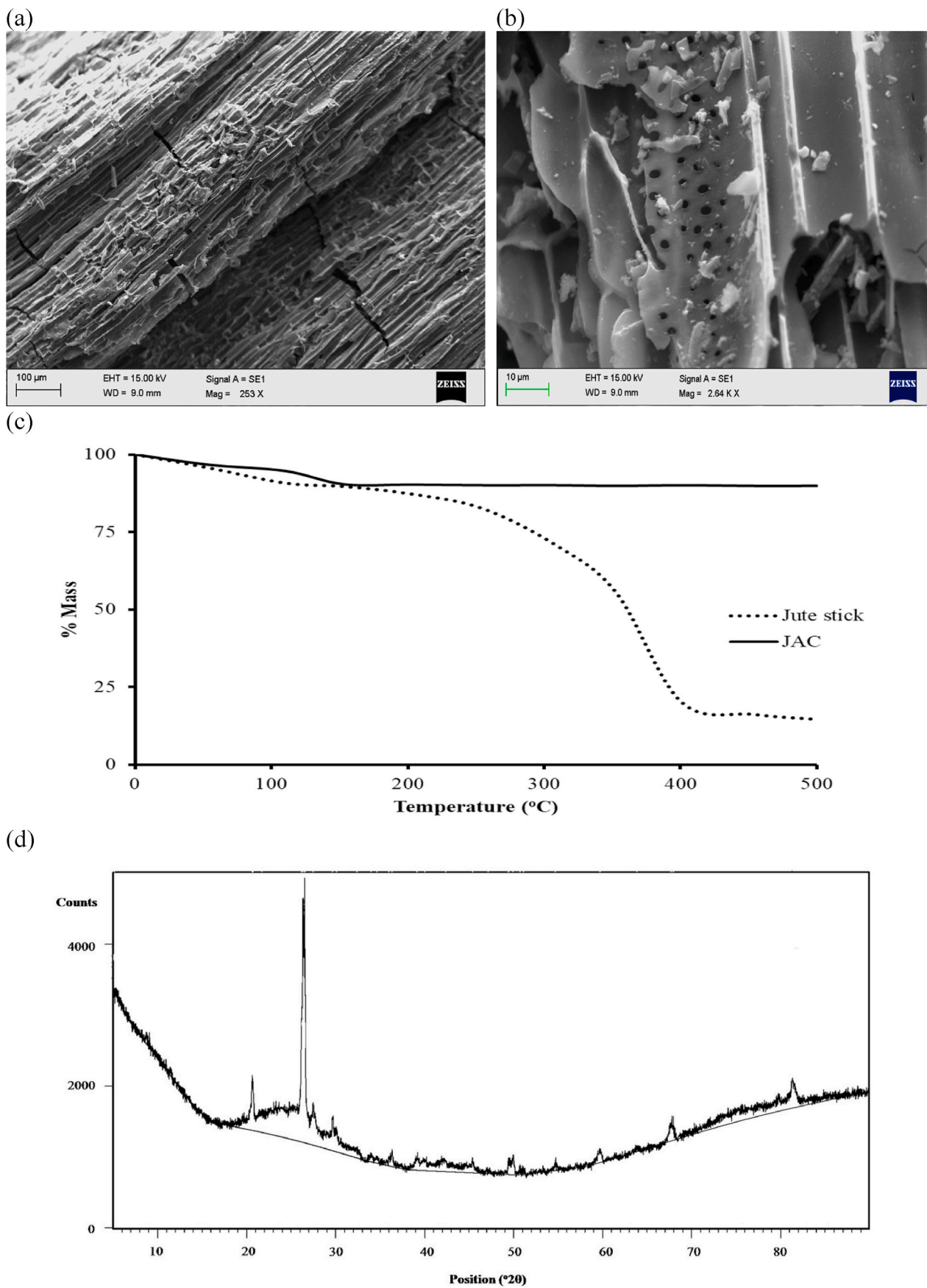
(a)

(b)

(c)

(d)

Fig. 1. Characterisation with SEM (a-jute stick; b- JAC), TGA (c) and XRD (d) analysis.

as a porous carbon material.

The BET (Brunauer-Emmett-Teller) surface area of JAC was 335 m^2/g, which was lower than the earlier reported jute carbon materials (730–1626 m^2/g). This might have happened because of a lower proportion (1:10) of H$_3$PO$_4$ used in the activation step in comparison with the previously published reports (ranging from 1:1 to 1:4) (Asadullah et al., 2010, 2014, 2014; Ghosh et al., 2020).

Here, the thermogravimetric analysis reports showed a contrasting thermal behaviour of jute stick vis-à-vis JAC (Fig. 1c). At various temperature zones, the former revealed three distinct regions of reaction. A mass loss of around 13.7% was observed, when the temperature was raised to 200 °C. This could be perhaps because of the loss of physically bound water. There was an additional mass loss of 67.1% within the temperature range of 200–400 °C, resulting from the degradation of cellulose and hemicellulose (Yang et al., 2007). At a higher temperature (above 400 °C), the lignin components were degraded, which finally generated around 14.7% of the residual mass (Raveendran et al., 1991). Contrastingly, the latter showed only a mass loss of 10.1% up to 200 °C. The hygroscopicity was due to the presence of various oxygenated functional groups, which retained the water molecules by hydrogen bonding. There was no significant mass loss between 200 °C and 500 °C. As demonstrated in an earlier study by Oliveira et al. (2017), around 89.9% residual mass yield was noted in JAC, suggesting the presence of

thermostable compounds.

The X-ray diffraction analysis of JAC exhibited a broad diffraction band at round 2θ = 22-30° along with some sharp peaks at 2θ maxima of 26 and 29°, which were associated with the diffraction of 002 plane (Fig. 1d). The corresponding mean d$_{002}$ values were 3.36 and 3.01 Å for 2θ maxima at 26° and 29° respectively. Notably, these peaks showed strong intensities (>80%), and the main polymeric chain of cellulose remained less affected during chemical activation with H$_3$PO$_4$ (Girgis et al., 2007). The diffraction around 2θ = 43° showed a weak, diffused and broad band with lesser developed peaks, which might have resulted from the poorly developed intragraphitic layers. The low diffraction peaks were also present at around 2θ = 14-22°, which might be caused by a partial destruction of the cellulose crystalline structure. Earlier, similar observations were reported in case of other agroresidue-derived activated carbons (Yun et al., 2001).

The FTIR analysis revealed the presence of various functional groups in jute stick (Fig. 2a). A broad band at around 3400-3200 cm^{-1} with a peak maxima at 3313 cm^{-1} and a small shoulder peak at 604 cm^{-1} represented the stretching vibrations of free –OH and out-of-plane deformation vibrations of –OH groups of cellulose and lignin. The bands appearing at around 2852 cm^{-1} were attributed to –CH stretching of methyl (-CH$_3$) and methylene (-CH$_2$) groups. The sharp peaks at 1734 cm^{-1} represented the –CO stretching vibrations of acetyl groups in
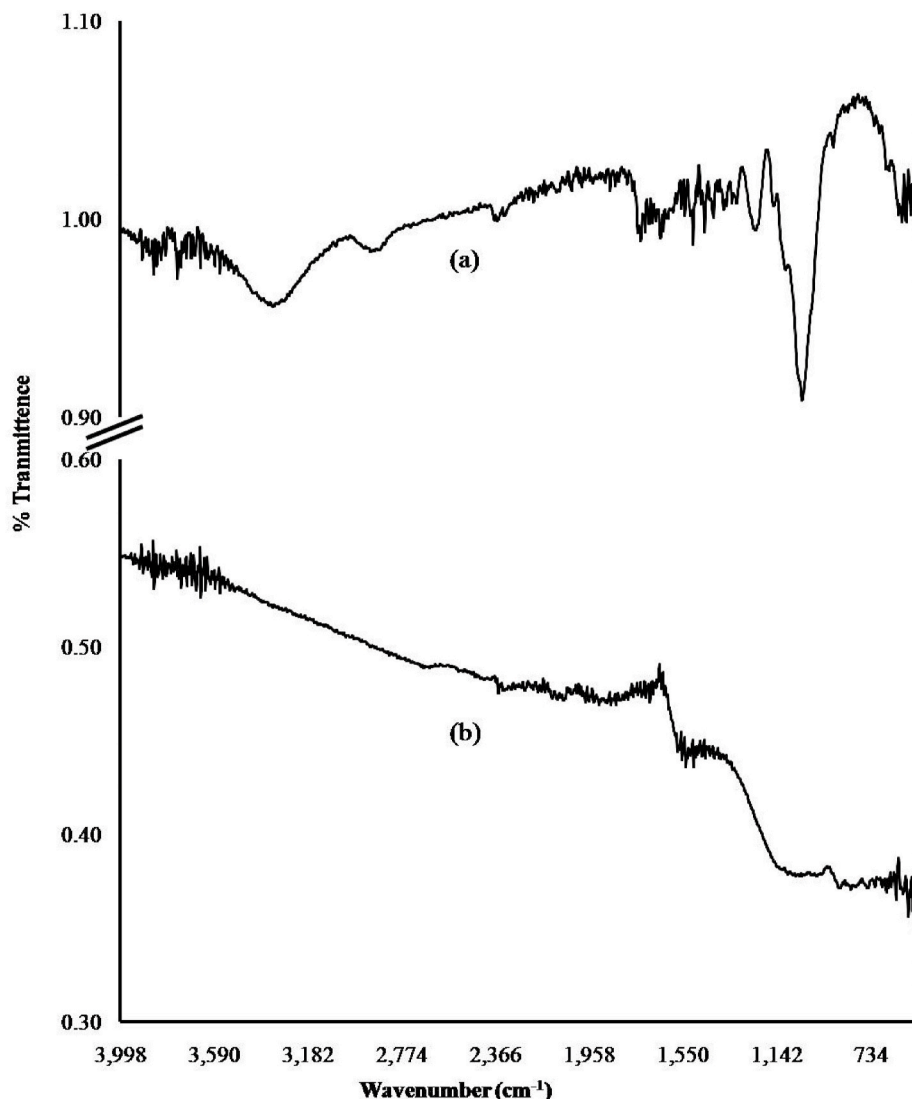

Fig. 2. Functional group analysis of (a) jute stick and (b) JAC by FTIR.

hemicellulose. A series of strong peaks at around 1600-1400 cm^{-1} (1636, 1506, 1456 and 1418 cm^{-1}) showed many aromatic ring (-C=C-) structures in jute stick. A clear peak at 1363 cm^{-1} represented the symmetric vibrations of in-plane deformation of a methyl (–CH$_3$) group, while a peak at 1224 cm^{-1} indicated the asymmetric = C–O–C stretching vibrations associated with aryl groups of lignin (Ghosh et al., 2020). Another clear peak at 1316 cm^{-1} was associated with O–H in-plane bending vibrations or C–O stretching vibrations of cellulose. Whereas, a weak signal at 895 cm^{-1} could be due to the C–H in-plane bending vibrations or C–H/O–H out-of-plane deformation vibrations in cellulose. These findings indicate the presence of the above-stated functional groups in the pyranoid rings of cellulose, which was also previously reported by Reddy et al. (2017). A strong peak at 1338 cm^{-1} corresponded to N–O stretching vibration, whereas, another at 1028 cm^{-1} demonstrated the stretching vibrations of C–O–C in the cellulose and lignin components of jute stick. The FTIR analysis clearly showed the transformation of various functional groups after the processing of jute stick into JAC (Fig. 2b). The fused bands seen with a poor peak resolution at around 3200 cm^{-1} suggests the presence of –OH group, which could be associated with phenols, alcohols, acids and adsorbed water.

The removal of characteristic peaks at around 2900-2800 cm^{-1} (for –CH$_3$ and –CH$_2$), 1734 cm^{-1} (for C=O), 1636 and 1456 cm^{-1} (for aromatic C=C) and 1338 cm^{-1} (for N–O stretching) demonstrated the decomposition and transformation of initial cellulose and hemicellulose of jute stick. As seen, the formation of some new bonds corresponding to the peaks at 1695 cm^{-1} hint at the presence of residual aldehydes or ketones, while the peaks at 1552 cm^{-1} suggest the presence of residual aromatic rings in the carbon layer. Besides, a very strong broad band with its maxima was also present at 1152 cm^{-1} (which was between 1300 and 900 cm^{-1}), suggesting the peaks of JAC. Several functional groups of jute stick was degraded during the carbonisation process that was indicated by the shifting of signals under the broad peak at around 1300-900 cm^{-1}. These were attributed to oxidised carbons including C–O in acids, esters, ethers and phenols (Yaket and El-Deen, 2016). Furthermore, the characteristic peaks at around 1200-1150 cm^{-1} for the stretching vibrations of H-bonded P=O, C–O of aromatic P–C–O and P=OOH from numerous phosphor-carbonaceous products were also present in this region (Yaket and El-Deen, 2016; Puziy et al., 2002). The formation of new peaks at 895, 844 and 777 cm^{-1} indicates C–H out-of-plane vibrations from substituted benzene compounds in JAC. As it was a porous and thermostable substance containing various surface active functional groups, JAC could be used as a dSPE sorbent in the cleanup step of pesticide residue analysis.

Table 1
Removal of co-eluting matrix compounds by various dSPE sorbents.

Pesticide (P)	Matrix compound (MC)	RT (min)	Control matrix extract	Spiked matrix (20 ppb) extract		
	Okra matrix		PSA	PSA	PSA + JAC	PSA + GCB
Ethalfluralin	7-Hexadecenal, (Z)-	8.274	MC	MC	P	P
Sulfotep	Oleyl alcohol, trifluoroacetate	8.523	MC	P	P	P
Clomazone	Dichloroacetic acid, 2-tridecyl ester	9.456	MC	MC	P	P
Pyrimiphos methyl	Eicosane	11.263	MC	MC	P	P
Fenitrothion	Pentanoic acid, 2-ethylcyclohexyl ester	11.345	MC	MC	P	P
Malathion	i-Propyl 14-methyl-pentadecanoate	11.478	MC	MC	P	P
Metolachlor	2-cyclohexylethyl butyl ester	11.618	MC	MC	P	P
Quinalphos	1-Eicosanol	12.623	MC	MC	P	P
Tetrachlorvinphos	Methyl stearate	13.014	MC	MC	P	P
Mirex	Fumaric acid	17.548	MC	MC	P	P
Fenarimol	Alpha- Tocopherol	17.647	MC	MC	P	P
Pomegranate matrix						
Dichlorvos	2-Isobutylideneamino-3-methylbutyronitrile	4.218	MC	MC	P	P
Etridiazole	Caryophyllene oxide	6.27	MC	MC	P	P
Cycloate	Tumerone	8.214	MC	MC	P	P
Ethalfluralin	Cholest-4-en-3-one, 26-hydroxy-	8.274	MC	MC	P	P
Benfluralin	aR-Turmerone	8.49	MC	MC	P	P
alpha-HCH	Curlone	8.951	MC	MC	P	P
Atrazine	(6R,7R)-Bisabolone	9.403	MC	MC	P	P
Nitralin	beta.-Sitosterol	15.843	MC	MC	P	P
Spinach matrix						
3,4-Dichloroaniline	Caryophyllene	6.164	MC	MC	P	P
Pebulate	Linalylphenylacetate	6.337	MC	MC	P	P
Propoxur	Fumaric acid, hexadecylpropargyl ester	7.902	MC	MC	P	P
Diphenylamine	Octatriacontylpentafluoropropionate	8.149	MC	MC	P	P
Prodiamine	Isophytol	11.316	MC	MC	P	P
Quinalphos	Phytol	12.623	MC	MC	P	P
Procymidone	Trilinolein	12.714	MC	MC	P	P
Tetrachlorvinphos	Methyl stearate	13.014	MC	MC	P	P
Paclobutrazol	Ethanone,1-[3-(5-furan-2-yl-[1,3,4]oxadiazol-2-ylsulfanylmethyl)-2,4,6-trimethylphenyl]-	13.079	MC	MC	P	P
o,p'-DDD	Phytol acetate	13.813	MC	MC	P	P
Edifenphos	Cycloisolongifolene	15.223	MC	MC	P	P
Cyfluthrin-1	Methyl 5,11,14,17-eicosatetraenoate	18.718	MC	MC	P	P
Tea matrix						
Tetrahydrophthalimide	2,4-Di-tert-butylphenol	6.744	MC	MC	P	P
Profluralin	Methyl piperonylate, 2-hydroxy-	9.543	MC	MC	P	P
Endosulfan ether	Caffeine	10.529	MC	MC	P	P
Propanil	Theobromine	10.966	MC	MC	P	P
Pirimiphos methyl	1,6-Octadiene, 3-ethoxy-3,7-dimethyl-	11.263	MC	MC	P	P
Quinalphos	9,12-Octadecadienoic acid (Z,Z)-, methyl ester	12.623	MC	MC	P	P
Triazophos	Oleic anhydride	14.884	MC	MC	P	P

3.2. Optimisation of the cleanup process

So far, various combinations of sorbents including PSA, GCB and C18 (octadecylsilane) could effectively remove matrix co-extractives in the cleanup step. In the past, numerous researchers have used a combination of PSA and GCB as a sorbent mixture in their methods (Lawal et al., 2018; Narenderan et al., 2020; Samsidar et al., 2018). While optimising the cleanup in this study, two levels (5 and 10 mg/mL) of JAC and GCB (each with a fixed amount of PSA, 25 mg/mL) were evaluated. Once these sorbents were added into the sample extract, it was vortexed for 1 min and then centrifuged (2800×g) for 5 min, before injecting into GC-MS/MS. The results in Table 1 presents the effect of various sorbents on cleanup. The GC-MS full scan analysis showed higher background signals, where matrix compounds co-eluted with the MRM of many of the targeted pesticides. As noted, these matrix compounds posed major problems in the identification and quantification of the coeluting pesticide residues, also similar to the finding of Markovic et al., 2009).

Based on the NIST mass spectral library matching (above 80%), the co-eluting matrix compounds in okra included eicosane, 1-eicosanol, methyl stearate, γ-sitosterol, ethyl oleate, linoleic acid ethyl ester, fumaric acid, among others. They co-eluted with many of the targeted pesticides (Table 1), which was in agreement with an earlier report of Huang et al. (2017). The effect of various sorbents on the cleanup of different matrices at a specific retention time window can be viewed in Table 1. By using only PSA, several matrix compounds (MC in Table 1) appeared as the co-eluting signals. Whereas, the combinations of JAC + PSA and GCB + PSA significantly removed the matrix-related interferences. This, in turn, helped in the identification of the target pesticides (P in Table 1). In pomegranate, ar-turmerone, curlone, β-sitosterol, (6R, 7R)-bisabolone, caryophyllene oxide and 26-hydroxy-cholest-4-en-3-one were the major interfering compounds. In spinach, the list included caryophyllene, phytol, cycloisolongifolene and methyl stearate, the signals of which interfered with 3,4-dichloroaniline, quinalphos, edifenphos and tetrachlorvinphos respectively. In case of tea,
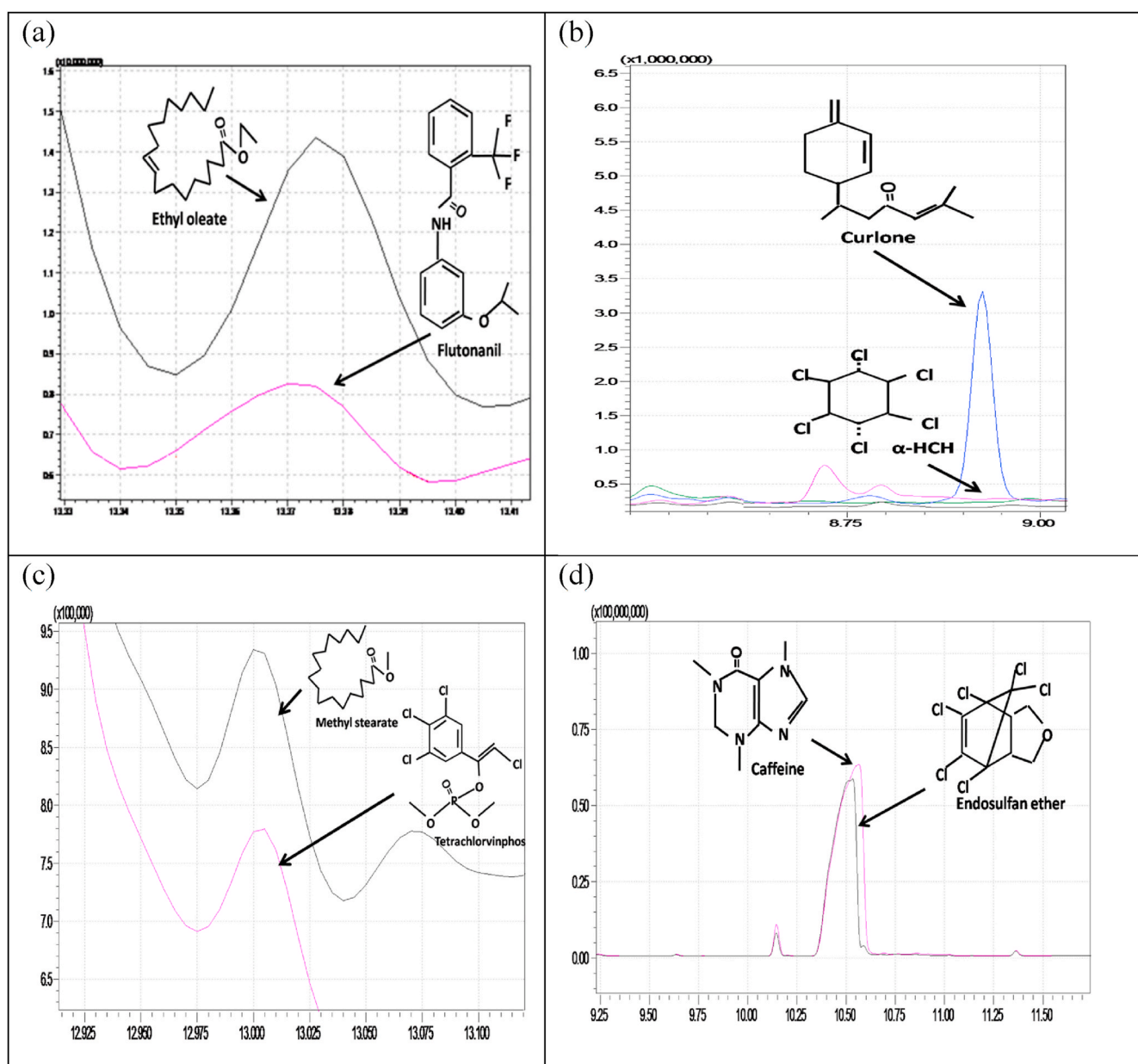

Fig. 3. Co-elution of matrix compounds with pesticides in (a) ethyl oleate with flutolanil in okra, (b) curlone with α-HCH in pomegranate, (c) methyl stearate with tetrachlorvinphos in spinach, and (d) caffeine with endosulfan ether in tea.

compounds such as caffeine, theobromine, 9,12-octadecadienoic acid (Z,Z)-, methyl ester and oleic anhydride exerted a higher matrix effect on endosulfan ether, propanil, quinalphos and triazophos respectively. In Fig. 3, the coelution of a few matrix compounds including ethyl oleate with flutolanil (R_t = 13.356 min) in okra, curlone with α-HCH (R_t = 8.951 min) in pomegranate, methyl stearate with tetrachlorvinphos (R_t = 13.014 min) in spinach and caffeine with endosulfan ether (R_t = 10.529 min) in tea were noted. These co-eluted matrix-derived compounds were responsible for the matrix effects on the corresponding pesticides, aligning with many previous studies (Khan et al., 2014; Ghosh et al., 2019; Islam et al., 2019). For effectively removing the coeluting matrix compounds from the test matrices, a combination of JAC with PSA or GCB with PSA (25 mg/mL extract) was required (Table 1).

In all the test compounds, a good linearity ($r^2 \geq 0.99$) was recorded for both the solvent and matrix-matched calibrations. In most cases, the LOQ was 10 ng/g. For both JAC and GCB cleanups, the recoveries with 5 mg/mL demonstrated satisfactory results in okra (Supplementary Table 1). Nevertheless, the recoveries of some pesticides were compromised at 10 mg/mL. In okra, 5 (chlorothalonil, cyprodinil, chlozolinate, propargite and azinphos-ethyl) and 14 (biphenyl, 3,4-dichloroaniline, diphenylamine, 2,3,5,6-tetrachloroaniline, hexachlorobenzene, tetrachlorobenzene, dicloran, pyrimethanil, chlorothalonil, 4,4′-dichlorobenzophenone, cyprodinil, chlozolinate, propargite and azinphos-ethyl) compounds showed no recoveries, especially when the cleanup involved dSPE with 10 mg/mL of JAC and GCB respectively. Many of these pesticides (*e.g.* chlorothalonil, cyprodinil, azinphos-ethyl, etc.) were earlier reported for a loss in recoveries, particularly when GCB was used for cleanup (Mol et al., 2007). Additionally, a significant loss in recoveries was recorded in the pesticides with a planar structure (Fig. 4), when the sorbent amount was increased from 5 mg/mL to 10 mg/mL. These findings indicate that recoveries of the number of pesticides with JAC-cleanup were higher than the GCB-based cleanup. The highest cleanup performance was obtained with 5 mg/mL of JAC. The sample extract resulting from the JAC-based cleanup appeared clearer than the GCB-cleaned extract, suggesting an effective removal of the co-extracted plant pigments. With the JAC-based cleanup, there were 49.2% and 54.8% reductions in the absorbance of chlorophyll A and B respectively, while the corresponding reductions were 75.6% and 65% respectively in case of GCB. The findings suggest a superior cleanup performance of JAC over GCB.

The gravimetric analysis also demonstrated a better performance of JAC as against GCB. The application of PSA (25 mg) as a sole adsorbent in the dSPE step reduced the residual matter by 8.3–10%. The addition of 5 mg of GCB along with PSA reduced the residues to the tune of
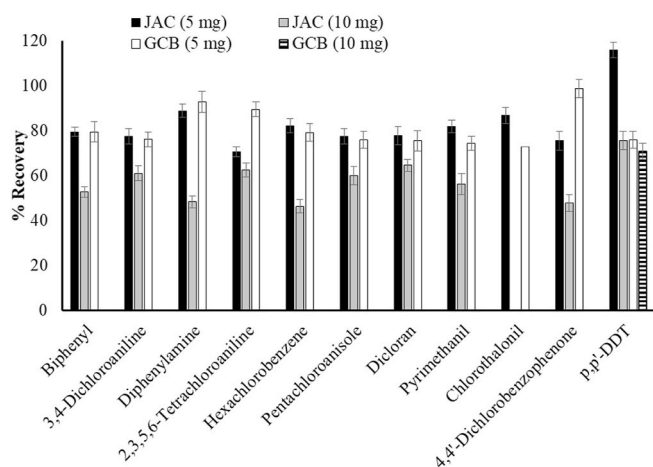
16.3–20%, while it was reduced by 40–46.7% for JAC, which further established a superior cleanup efficiency of JAC. After the JAC and GCB-based cleanup, the pesticide recoveries in various matrices were in the range of 63.2–120% and 69–127.5% respectively in okra, 62–117.9% and 60.4–117% respectively in pomegranate, 68.6–126.8% and 61–114.6% respectively in spinach and 69.3–117.4% and 59.8–113.9% respectively in tea (Supplementary Table 2). All these recoveries were within the acceptable range of 70–120% (SANTE, 2019). The JAC treatment (at 5 mg/mL) resulted in significantly higher recoveries for 74 compounds in okra, 70 compounds in pomegranate, 70 compounds in spinach and 80 compounds in tea compared to the GCB treatment. In some cases, both JAC and GCB cleanups resulted in similar recoveries (for 48 compounds in okra, 40 compounds in pomegranate, 47 compounds in spinach and 40 compounds in tea).

3.3. Matrix effect (ME)

To understand the matrix effects (MEs), the GC-MS/MS chromatograms were virtually divided into four R_t (min) windows of 6.01–10.00 min, 10.53–13.00 min, 13.1–16.0 min and 16.01–20.0 min. Based on earlier reports (Chatterjee et al., 2016; Islam et al., 2019), the ME (%) results were grouped into five categories as follows: high signal suppression (ME < −50%), moderate signal suppression (ME = −20 to −50%), no matrix effect (ME = −20 to 20%), moderate signal enhancement (ME = 20–50%) and high signal enhancement (ME > 50%). With the treatment of 5 mg/mL of JAC and GCB (Table 2, Fig. 5), the ME appreciably varied in all matrices. In case of JAC, the ME was nominal for the following number of pesticides: 50 (27.6%) in okra, 87 (48.1%) in spinach, 113 (62.4%) in pomegranate and 137 (75.7%) in tea. The number of pesticides with 'no matrix effect' in JAC was considerably higher than GCB. These included 7 compounds (3.9%) in okra, 84 (46.4%) compounds in spinach, 45 compounds (24.9%) in pomogranate and 33 compounds (18.7%) in tea (Fig. 5). Compared to the GCB treatment, the JAC-based cleanup resulted in a lower matrix effect. In okra, for example, 90 (49.7%) and 103 (56.9%) pesticides showed a moderate signal enhancement (>20% and <50%) for JAC and GCB respectively. Such kind of moderate signal enhancement was also observed in the remainder matrices for the JAC and GCB cleanups: 41 (22.6%) and 52 (28.7%) pesticides respectively in spinach, 66 (36.4%) and 117 (64.6%) pesticides respectively in pomegranate and 39 (21.5%) and 135 (74.8%) pesticides respectively in tea. After the JAC and GCB treatments, a higher signal enhancement was observed for 40 (22.1%) and 70 (38.7%) pesticides respectively in okra, 28 (15.5%) and 32 (17.7%) pesticides respectively in spinach, 2 (1.1%) and 19 (10.5%) pesticides respectively in pomegranate and 5 (2.8%) and 13 (7.2%) pesticides respectively in tea.

In comparison with GCB, the use of JAC resulted in a better signal to noise ratio (S/N) for most of the compounds. For example, when the okra extract was treated with GCB, the S/N of chlorothalonil, biphenyl, dicloran, clomazone, chlorpyrifos methyl, diphenyl amine and tetrachlorvinphos were 1248.11, 1474.8, 79.2, 3066, 1740.3, 695.3 and 1076 respectively. Whereas, with the JAC treatment, a better S/N was obtained in chlorothalonil (S/N = 1503.88), biphenyl (S/N = 3091), dicloran (S/N = 1416.2), clomazone (S/N = 4596), chlorpyrifos methyl (S/N = 3053.89), diphenyl amine (S/N = 2308) and tetrachlorvinphos (S/N = 1299). However, the signal suppressions were insignificant in okra, pomegranate and tea with JAC. A few cases of peak suppression were recorded too in spinach. For spinach, only 8.3% (JAC) and 3.9% (GCB) suffered from a moderate signal suppression. A higher signal suppression (above 50%) was recorded for 5.5% (JAC) and 3.1% (GCB) pesticides. These results established a superior cleanup effect of JAC over GCB.

Thus, JAC demonstrated a comparable performance with other adsorbents in terms of the rate of use and suitability for the multiresidue analysis of pesticides. The comparative cleanup effects of GCB and non-GCB sorbents vis-à-vis the newly developed JAC are presented in

Fig. 4. Effect of dSPE applications (@ 5 and 10 mg/mL of JAC and GCB) on the recovery of planar pesticides in okra.

Table 2
Matrix effect on cleanup with JAC (5 mg/mL) and GCB (5 mg/mL).

Compound Name	Rt (min)	Okra		Spinach		Pomegranate		Tea	
		JAC	GCB	JAC	GCB	JAC	GCB	JAC	GCB
Dichlorvos	4.218	26.9 [a]	35.5 [b]	20.2 [a]	26.1 [b]	20.9 [a]	33.4 [b]	10.4 [a]	23.1 [b]
4-Bromo-2-chlorophenol	4.56	31.5 [a]	38.1 [b]	−20.9 [b]	−0.40 [a]	12.3 [a]	15.6 [a]	8.40 [a]	23.9 [b]
Allidochlor	4.644	10.6 [a]	23.6 [b]	1.90 [a]	10.4 [b]	14.3 [a]	17.6 [b]	12.6 [a]	22.6 [b]
Biphenyl	5.673	6.40 [a]	20.7 [b]	5.30 [a]	10.5 [b]	26.6 [a]	29.6 [b]	19.1 [a]	23.5 [b]
3,4-Dichloroaniline	6.164	30.3 [a]	42.9 [b]	28.9 [a]	38.7 [b]	11.9 [a]	15.4 [b]	4.50 [a]	19.2 [b]
Etridiazole	6.27	40.5 [a]	46.0 [b]	28.8 [a]	38.7 [b]	25.0 [a]	28.7 [b]	4.50 [a]	19.2 [b]
Pebulate	6.337	10.7 [a]	22.6 [b]	−46.6 [b]	−34.6 [a]	21.5 [a]	35.6 [b]	10.7 [a]	24.7 [b]
Methacrifos	6.689	23.5 [a]	35.5 [b]	−54.1 [b]	−32.0 [a]	16.2 [a]	27.2 [b]	17.1 [a]	23.9 [b]
Tetrahydrophthalimide	6.744	58.8 [a]	68.6 [b]	−13.2 [b]	−3.70 [a]	18.7 [a]	19.8 [a]	4.20 [a]	17.3 [b]
2-Phenylphenol	7.052	30.9 [a]	39.7 [b]	−5.40 [a]	5.60 [a]	16.8 [a]	18.0 [a]	8.10 [a]	21.4 [b]
Pentachlorobenzene	7.073	9.25 [a]	23.4 [b]	72.9 [a]	66.4 [a]	18.5 [a]	19.4 [b]	13.9 [a]	29.4 [b]
Tecnazene	7.792	50.1 [a]	58.9 [b]	−16.6 [b]	−4.20 [a]	17.5 [a]	20.4 [b]	8.50 [a]	24.4 [b]
Propachlor	7.902	29.9 [a]	40.9 [b]	−19.1 [b]	−2.70 [a]	29.0 [a]	34.3 [b]	4.30 [a]	20.4 [b]
Diphenylamine	8.149	11.3 [a]	24.6 [b]	−30.0 [b]	−20.9 [a]	16.6 [a]	19.3 [b]	16.3 [a]	25.8 [b]
2,3,5,6-Tetrachloroaniline	8.189	11.7 [a]	24.1 [b]	7.00 [a]	19.4 [b]	27.2 [a]	27.3 [a]	6.90 [a]	20.0 [a]
Cycloate	8.214	9.90 [a]	23.9 [b]	42.6 [a]	48.9 [b]	18.6 [a]	41.0 [b]	8.40 [a]	18.6 [b]
Ethalfluralin	8.274	63.8 [a]	68.1 [b]	−22.7 [b]	−9.90 [a]	23.2 [a]	24.5 [a]	7.20 [a]	19.4 [b]
Chlorpropham	8.429	30.6 [a]	44.5 [b]	−13.1 [b]	−1.00 [a]	20.0 [a]	28.6 [b]	19.0 [a]	27.9 [b]
Trifluralin	8.434	58.3 [a]	65.7 [b]	−4.10 [a]	7.40 [b]	14.8 [a]	14.9 [a]	12.5 [a]	20.2 [b]
Benfluralin	8.49	57.2 [a]	63.9 [b]	−3.10 [a]	6.60 [b]	20.4 [a]	20.8 [a]	14.1 [a]	27.2 [b]
Sulfotep	8.523	30.4 [a]	42.6 [b]	−18.9 [b]	−6.20 [a]	20.7 [a]	21.2 [a]	16.1 [a]	24.8 [b]
Bendiocarb	8.529	53.8 [a]	53.8 [a]	−5.60 [a]	19.1 [b]	17.6 [a]	96.0 [b]	6.80 [a]	16.1 [b]
Di-allate-1	8.782	8.63 [a]	25.2 [b]	−14.9 [b]	−0.50 [a]	36.5 [a]	44.0 [b]	16.3 [a]	23.2 [b]
Phorate	8.789	49.8 [a]	49.1 [a]	−5.40 [a]	5.60 [b]	20.3 [a]	26.4 [b]	11.8 [a]	23.1 [b]
alpha-HCH	8.951	13.8 [a]	27.4 [b]	55.5 [a]	61.1 [a]	31.6 [a]	37.1 [b]	9.40 [a]	23.0 [b]
Hexachlorobenzene	9.052	13.0 [a]	25.7 [b]	−14.5 [b]	−4.50 [a]	24.1 [a]	56.1 [b]	12.4 [a]	25.3 [b]
Pentachloroanisole	9.145	12.6 [a]	27.3 [b]	−1.70 [a]	9.90 [b]	22.6 [a]	23.1 [a]	13.0 [a]	22.5 [b]
Dicloran	9.228	53.6 [a]	62.5 [b]	−11.6 [b]	−1.70 [a]	15.8 [a]	19.8 [b]	11.2 [a]	22.1 [b]
Atrazine	9.403	26.3 [a]	35.7 [b]	−7.60 [a]	−5.70 [a]	25.9 [a]	28.1 [a]	8.60 [a]	23.3 [b]
Clomazone	9.456	21.2 [a]	32.1 [b]	−60.0 [b]	−16.4 [a]	17.0 [a]	23.8 [b]	9.40 [a]	21.1 [b]
Profluralin	9.543	65.4 [a]	69.6 [a]	−8.10 [a]	1.40 [b]	19.4 [a]	24.9 [b]	16.5 [a]	26.3 [b]
gamma-HCH (Lindane)	9.625	11.8 [a]	21.3 [b]	41.4 [a]	47.8 [a]	34.0	42.9	8.10	20.8
Pentachlorobenzonitrile	9.63	14.7 [a]	28.4 [b]	−12.6 [a]	4.80 [b]	18.7 [a]	18.8 [a]	12.5 [a]	24.3 [b]
Terbufos	9.639	68.5 [a]	75.4 [b]	−6.60 [a]	6.60 [b]	22.9 [a]	23.3 [a]	13.5 [a]	20.9 [b]
Terbuthylazine	9.671	12.0 [a]	30.4 [b]	−15.1 [b]	−1.10 [a]	18.8 [a]	23.2 [b]	18.7 [a]	38.4 [b]
Diazinon	9.741	31.6 [a]	47.3 [b]	−6.30 [a]	2.60 [b]	20.4 [a]	24.2 [b]	14.1 [a]	25.4 [b]
Fonofos	9.748	21.9 [a]	35.6 [b]	−23.7 [b]	5.30 [a]	21.1 [a]	27.8 [b]	11.4 [a]	26.4 [b]
Propyzamide	9.756	20.7 [a]	35.1 [b]	16.2 [a]	24.1 [b]	12.2 [a]	24.5 [b]	2.70 [a]	15.8 [b]
Fluchloralin	9.801	59.5 [a]	68.4 [b]	−6.10 [a]	2.20 [b]	10.7 [a]	20.7 [b]	3.50 [a]	22.6 [b]
Pyrimethanil	9.911	1.34 [a]	27.3 [b]	−40.9 [b]	−16.6 [a]	10.3 [a]	14.9 [b]	17.8 [a]	28.6 [b]
Chlorothalonil	10.00	25.2 [a]	28.7 [a]	−26.5 [b]	−17.0 [a]	9.90 [a]	23.2 [b]	0.10 [a]	15.5 [b]
Isazofos	9.99	37.7 [a]	49.2 [b]	1.40 [a]	8.20 [b]	24.5 [a]	29.9 [b]	16.9 [a]	27.4 [b]
Tefluthrin	10.004	12.6 [a]	26.3 [b]	−29.9 [b]	−6.50 [a]	9.70 [a]	27.6 [b]	18.3 [a]	27.1 [b]
Terbacil	10.084	64.6 [a]	70.3 [a]	−14.3 [b]	−7.20 [a]	17.3 [a]	23.5 [b]	5.30 [a]	21.3 [b]
Etrimfos	10.082	36.8 [a]	47.0 [b]	44.8 [a]	53.2 [b]	20.9 [a]	21.7 [a]	12.9 [a]	23.5 [b]
Tri-allate	10.148	23.1 [a]	34.8 [b]	25.9 [a]	31.9 [b]	6.60 [a]	14.4 [b]	14.1 [a]	25.2 [b]
Disulfoton	10.217	63.3 [a]	71.3 [b]	48.2 [a]	55.9 [b]	17.8 [a]	20.7 [b]	84.2 [a]	86.2 [a]
Endosulfan ether	10.529	54.8 [b]	28.6 [a]	11.6 [a]	23.4 [b]	12.2 [a]	19.8 [b]	3.70 [a]	15.4 [b]
Pentachloroaniline	10.543	9.70 [a]	22.4 [b]	58.6 [a]	55.7 [a]	11.7 [a]	18.0 [b]	10.3 [a]	23.9 [b]
Dimethachlor	10.615	47.4 [a]	56.4 [b]	−12.2 [b]	−6.00 [a]	19.5 [a]	22.8 [b]	18.8 [a]	28.3 [b]
Acetochlor	10.696	46.6 [a]	52.5 [b]	−20.2 [b]	−10.0 [a]	20.7 [a]	24.3 [b]	11.3 [a]	23.9 [b]
Propanil	10.966	17.1 [a]	19.7 [a]	−4.90 [a]	13.3 [b]	19.9 [a]	29.8 [b]	20.2 [a]	37.6 [b]
Chlorpyrifos-methyl	10.738	27.6 [a]	44.1 [b]	27.6 [a]	38.7 [b]	10.7 [a]	16.7 [b]	38.9 [b]	33.4 [a]
Vinclozolin	10.813	7.40 [a]	27.1 [b]	10.9 [a]	20.2 [b]	22.4 [a]	26.5 [b]	8.20 [a]	26.6 [b]
Alachlor	10.873	44.8 [a]	54.7 [b]	27.6 [a]	38.7 [b]	17.1 [a]	20.9 [b]	8.10 [a]	21.8 [b]
Tolclofos-methyl	10.883	35.4 [a]	38.4 [a]	−6.80 [a]	8.30 [b]	10.3 [a]	15.5 [b]	12.3 [a]	15.6 [b]
Transfluthrin	10.894	24.5 [a]	31.4 [b]	8.40 [a]	24.9 [b]	20.8 [a]	29.0 [b]	12.0 [a]	18.2 [b]
Propisochlor	10.954	51.4 [a]	58.3 [b]	−6.80 [a]	8.30 [b]	19.1 [a]	30.3 [b]	12.2 [a]	14.8 [b]
Metalaxyl (Mefenoxam)	10.997	34.4 [a]	45.4 [b]	−1.90 [a]	13.9 [b]	13.2 [a]	20.3 [b]	6.00 [a]	23.7 [b]
Heptachlor	11.06	78.7 [a]	82.5 [a]	−3.80 [b]	0.30 [a]	50.5 [a]	62.3 [b]	26.7 [a]	36.5 [b]
Fenchlorphos	11.08	32.7 [a]	43.2 [b]	9.90 [a]	28.2 [b]	14.4 [a]	19.4 [b]	7.90 [a]	25.7 [b]
Pirimiphos-methyl	11.263	27.2 [a]	44.1 [b]	−3.10 [a]	8.80 [b]	10.0 [a]	11.4 [a]	14.3 [b]	7.60 [a]
Prodiamine	11.316	69.1 [a]	74.8 [a]	19.1 [a]	23.8 [b]	15.4 [a]	27.6 [b]	8.70 [a]	23.0 [b]
Fenitrothion	11.345	17.3 [a]	64.3 [b]	5.24 [a]	25.4 [b]	24.5 [a]	29.6 [b]	60.6 [a]	57.6 [a]
Malathion	11.478	53.8 [a]	58.9 [a]	−15.9 [a]	−0.40 [a]	18.1 [a]	21.5 [b]	10.3 [a]	20.7 [b]
Dichlofluanid	11.486	39.5 [a]	41.7 [b]	−3.10 [a]	8.80 [b]	22.6 [a]	28.7 [b]	24.8 [a]	27.3 [a]
Pentachlorothioanisole	11.508	14.6 [a]	25.2 [b]	16.0 [a]	26.7 [b]	23.4 [a]	26.7 [b]	19.5 [a]	22.8 [b]
Metolachlor	11.618	21.7 [a]	52.4 [b]	8.20 [a]	18.5 [b]	9.70 [a]	15.1 [b]	15.9 [a]	15.4 [a]
Chlorpyrifos	11.653	19.5 [a]	48.9 [b]	22.5 [a]	23.3 [a]	17.3 [a]	21.1 [b]	25.0 [b]	8.70 [a]
Fenthion	11.734	56.3 [a]	67.4 [b]	24.1 [a]	39.5 [b]	17.7 [a]	19.8 [a]	13.4 [a]	14.8 [a]
Chlorthal-dimethyl	11.771	16.9 [a]	33.2 [b]	8.20 [a]	18.5 [b]	17.1 [a]	22.2 [b]	18.4 [a]	24.5 [b]
Aldrin	11.511	19.6 [a]	32.2 [b]	18.9 [b]	15.3 [a]	21.9 [a]	29.1 [b]	22.4 [a]	26.5 [b]
Anthraquinone	11.85	24.9 [b]	12.4 [a]	29.6 [a]	37.4 [b]	18.8 [a]	19.5 [a]	17.1 [a]	20.9 [b]

(*continued on next page*)

Table 2 (*continued*)

Compound Name	Rt (min)	Okra		Spinach		Pomegranate		Tea	
		JAC	GCB	JAC	GCB	JAC	GCB	JAC	GCB
Triadimefon	11.856	26.6 [a]	25.6 [a]	22.5 [a]	23.3 [a]	21.2 [a]	39.1 [b]	12.5 [a]	13.6 [a]
4,4′-Dichlorobenzophenone	11.997	19.0 [a]	−65.7 [b]	20.3 [a]	26.3 [b]	13.5 [a]	18.6 [b]	20.8 [a]	29.0 [b]
Pirimiphos ethyl	12.034	41.3 [a]	46.0 [b]	7.10 [a]	11.6 [b]	17.4 [a]	35.5 [b]	26.9 [a]	28.5 [b]
Fenson	12.087	14.2 [a]	27.6 [b]	33.8 [a]	43.8 [b]	19.4 [a]	28.7 [b]	19.1 [a]	30.3 [b]
Bromophos	12.105	32.4 [a]	47.2 [b]	−3.40 [a]	3.70 [b]	19.9 [a]	29.8 [b]	13.2 [a]	20.3 [b]
MGK 264-1	12.113	19.5 [a]	29.3 [b]	9.50 [a]	13.6 [b]	12.0 [a]	19.7 [b]	50.5 [a]	52.3 [a]
Isopropalin	12.151	58.2 [a]	68.1 [b]	−21.1 [b]	−5.00 [a]	15.3 [a]	20.5 [b]	14.4 [a]	19.4 [b]
Fipronil sulphide	12.21	40.1 [a]	44.4 [a]	20.9 [a]	41.3 [b]	18.1 [a]	29.0 [b]	19.4 [a]	24.9 [b]
Pendimethalin	12.309	64.6 [a]	69.3 [a]	−7.60 [b]	4.40 [a]	24.5 [a]	44.2 [b]	15.4 [a]	27.6 [b]
Isodrin	12.347	21.9 [a]	25.8 [b]	−21.9	−0.40 [a]	17.0 [a]	19.7 [b]	18.1 [a]	21.5 [b]
Metazachlor	12.353	60.2 [a]	64.1 [a]	−2.30 [a]	−2.10 [a]	13.4 [a]	22.0 [b]	22.6 [a]	28.7 [b]
Cyprodinil	12.354	23.1 [a]	30.0 [b]	10.3 [a]	22.1 [b]	21.1 [a]	38.7 [b]	17.3 [a]	44.7 [b]
MGK 264-2	12.357	14.3 [a]	26.5 [b]	−2.50 [a]	8.90 [b]	20.5 [a]	23.3 [b]	18.3 [a]	22.5 [b]
Fipronil	12.423	52.0 [a]	64.2 [b]	59.1 [b]	27.1 [a]	17.4 [a]	26.6 [b]	18.6 [a]	26.9 [b]
Chlozolinate	12.437	31.5 [a]	45.6 [b]	−9.80 [b]	2.90 [a]	9.90 [a]	16.8 [b]	7.20 [a]	19.2 [b]
Penconazole	12.443	24.8 [a]	41.1 [b]	−4.10 [b]	−0.60 [a]	25.6 [a]	27.2 [a]	8.20 [a]	20.1 [b]
(Z)-Chlorfenvinphos	12.483	52.6 [a]	63.6 [b]	23.7 [a]	35.0 [b]	21.8 [a]	24.2 [a]	99.8 [a]	95.8 [a]
Heptachlor epoxide	12.526	42.1 [a]	43.6 [a]	97.3 [a]	96.2 [a]	20.9 [a]	17.3 [a]	61.5 [b]	85.6 [b]
Quinalphos	12.623	19.7 [a]	50.6 [b]	20.8 [a]	25.2 [b]	6.60 [a]	15.0 [b]	24.2 [a]	31.2 [b]
Procymidone	12.714	20.0 [a]	29.3 [b]	5.90 [a]	11.1 [b]	17.9 [a]	24.4 [b]	15.6 [a]	21.6 [b]
Triflumizole	12.715	41.5 [a]	50.8 [b]	−24.1 [b]	−11.7 [a]	16.9 [a]	31.4 [b]	35.1 [a]	40.6 [b]
Bromophos-ethyl	12.896	28.6 [a]	42.2 [b]	99.3 [a]	99.2 [a]	17.0 [a]	20.0 [b]	15.7 [a]	31.6 [b]
Chlorbenside	12.975	32.0 [a]	47.5 [b]	62.3 [a]	57.8 [a]	24.4 [a]	30.0 [b]	25.7 [a]	34.8 [b]
cis-Chlordane	12.991	15.3 [a]	26.2 [b]	37.1 [b]	32.3 [a]	23.8 [a]	40.0 [b]	21.2 [a]	28.7 [b]
trans-Chlordane	12.991	24.2 [a]	33.9 [b]	75.4 [a]	80.1 [b]	13.4 [a]	48.5 [b]	4.40 [a]	25.4 [b]
DDMU	13.011	10.2 [a]	15.6 [b]	51.3 [a]	55.4 [a]	14.7 [a]	19.2 [b]	20.8 [a]	27.8 [b]
o,p'-DDE	13.013	11.3 [a]	29.2 [b]	99.3 [a]	99.2 [a]	28.4 [a]	99.8 [b]	14.4 [a]	17.4 [b]
Tetrachlorvinphos	13.014	8.00 [a]	76.3 [b]	77.5 [a]	82.7 [a]	11.8 [a]	18.4 [b]	7.70 [a]	25.4 [b]
Paclobutrazol	13.079	−25.8 [a]	72.4 [b]	−11.6 [b]	−4.60 [a]	18.0 [a]	21.0 [b]	17.3 [a]	34.1 [b]
alpha-Endosulfan	13.249	1.50 [a]	5.90 [b]	55.0 [a]	54.4 [a]	31.7 [a]	49.2 [b]	15.4 [a]	21.0 [b]
Bromfenvinphos	13.261	57.8 [a]	65.1 [b]	−15.6 [b]	−1.20 [a]	16.0 [a]	34.3 [b]	8.80 [a]	24.9 [b]
Flutriafol	13.298	46.2 [a]	72.7 [b]	47.4 [a]	52.8	19.7 [a]	27.4 [b]	14.6 [a]	29.4 [b]
Fenamiphos	13.307	44.1 [a]	95.2 [b]	−16.7 [b]	5.80 [a]	17.7 [a]	32.5 [b]	20.1 [a]	33.0 [b]
Flutolanil	13.365	43.8 [a]	53.2 [b]	−11.7 [b]	1.10 [a]	14.8 [a]	19.4 [b]	15.8 [a]	17.5 [b]
Iodofenphos	13.421	41.8 [a]	52.8 [b]	42.3 [a]	46.9 [a]	25.2 [a]	26.2 [a]	15.3 [a]	72.1 [b]
Chlorfenson	13.439	13.1 [a]	−15.0[b]	77.5 [a]	82.7 [a]	15.1 [a]	30.6 [b]	7.10 [a]	23.0 [b]
Prothiofos	13.462	−10.7 [a]	18.0 [b]	−15.0 [b]	−1.20 [a]	16.7 [a]	21.4 [b]	20.9 [a]	24.1 [b]
Pretilachlor	13.471	59.2 [a]	64.0 [a]	−16.4 [a]	−18.1 [a]	20.4 [a]	30.0 [b]	26.8 [a]	29.8 [b]
Fludioxonil	13.522	44.5 [a]	57.2 [b]	−15.0 [b]	−1.20 [a]	28.1 [a]	28.2 [a]	20.9 [a]	24.1 [b]
Profenofos	13.544	65.4 [a]	69.2 [a]	39.4 [a]	44.3 [b]	15.5 [a]	23.7 [b]	37.2 [a]	40.5 [a]
Tricyclazole	13.565	48.1 [a]	87.5 [b]	17.5 [a]	27.1 [b]	16.1 [a]	50.0 [b]	20.3 [a]	28.8 [b]
Oxadiazon	13.607	19.3 [a]	30.2 [b]	55.7 [a]	58.2 [a]	16.3 [a]	24.6 [b]	6.60 [a]	22.6 [b]
p,p'-DDE	13.663	10.5 [a]	24.3 [b]	32.7 [a]	40.3 [b]	16.1 [a]	24.5 [b]	15.4 [a]	16.5 [a]
Oxyfluorfen	13.728	53.2 [a]	74.4 [b]	−17.1 [b]	−2.40 [a]	4.90 [a]	15.8 [b]	10.6 [a]	17.7 [b]
Myclobutanil	13.729	49.1 [a]	58.2 [b]	43.8 [a]	48.3 [a]	15.1 [a]	18.6 [b]	7.60 [a]	21.8 [b]
Bupirimate	13.75	26.1 [a]	45.4 [b]	−7.50 [b]	−0.90 [a]	11.4 [a]	99.8 [b]	23.9 [a]	27.9 [b]
Flusilazole	13.761	32.0 [a]	44.1 [b]	−3.90 [a]	5.80 [b]	29.6 [a]	38.6 [b]	13.9 [a]	26.5 [b]
o,p'-DDD	13.813	18.6 [a]	33.2 [b]	62.6 [a]	67.8 [a]	28.7 [a]	29.8 [a]	14.1 [a]	28.6 [b]
Dieldrin	14.112	27.1 [a]	39.9 [b]	−6.10 [a]	13.3 [b]	18.0 [a]	23.2 [b]	11.8 [a]	25.9 [b]
Fluazifop-P-butyl	14.112	36.2 [a]	48.1 [b]	2.30 [a]	14.1 [b]	19.5 [a]	28.0 [b]	12.1 [a]	23.5 [b]
1,1-Dichloro-2,2-bis(4-ethylphenyl)ethane	14.196	19.4 [a]	32.6 [b]	31.7 [a]	39.2 [b]	9.90 [a]	30.5 [b]	17.8 [a]	27.5 [b]
Chlorobenzilate	14.374	45.5 [a]	62.4 [b]	42.9 [a]	48.8 [b]	20.2 [a]	26.2 [b]	22.7 [a]	26.3 [b]
beta-Endosulfan	14.529	30.0 [a]	52.1 [b]	−10.7 [a]	15.6 [b]	15.1 [a]	28.6 [b]	38.5 [a]	42.6 [a]
Ethion	14.53	46.2 [a]	54.1 [b]	−16.7 [b]	−4.20 [a]	13.9 [a]	19.9 [b]	11.8 [a]	26.1 [b]
Chlorthiophos-2	14.544	27.2 [a]	41.4 [b]	−0.10 [a]	16.5 [b]	19.1 [a]	22.3 [b]	7.50 [a]	22.4 [b]
o,p'-DDT	14.614	23.9 [a]	34.6 [b]	−2.40 [a]	10.1 [b]	21.1 [a]	22.1 [a]	27.7 [a]	29.8 [b]
Chlorthiophos-3	14.611	32.4 [a]	44.5 [b]	6.60 [a]	18.4 [b]	11.4 [a]	20.0 [b]	10.9 [a]	33.9 [b]
p,p'-DDD	14.614	27.8 [a]	39.0 [b]	85.8 [a]	91.7 [a]	14.5 [a]	20.0 [b]	29.4 [a]	39.5 [b]
cis-Nonachlor	14.634	15.9 [a]	33.8 [b]	−29.1 [b]	2.80 [a]	23.8 [a]	26.3 [b]	36.2 [a]	67.3 [b]
Endrin aldehyde	14.849	−13.6[b]	6.00 [a]	6.50 [a]	13.9 [b]	10.7 [a]	24.6 [b]	16.7 [a]	19.5 [b]
Triazophos	14.884	40.8 [a]	73.2 [b]	−39.4 [b]	−26.9 [a]	20.4 [a]	65.3 [b]	14.7 [a]	17.5 [b]
Sulprofos	14.915	32.8 [a]	72.2 [b]	−18.4 [a]	−18.5 [a]	12.3 [a]	23.5 [b]	16.3 [a]	24.5 [b]
Carfentrazone-ethyl	15.038	46.9 [a]	62.7 [b]	4.20 [a]	33.5 [b]	16.2 [a]	99.7 [b]	16.7 [a]	18.9 [b]
4,4′-methoxychlor olefin	15.142	6.10 [a]	26.2 [b]	−5.70 [a]	7.40 [b]	9.80 [a]	15.5 [b]	16.9 [a]	29.1 [b]
Carbophenothion	15.158	49.7 [a]	57.3 [b]	15.7 [a]	29.7 [b]	10.1 [a]	28.6 [b]	27.3 [a]	37.3 [b]
Edifenphos	15.223	57.0 [a]	78.8 [b]	28.2 [a]	35.0 [b]	14.0 [a]	99.3 [b]	14.9 [a]	16.7 [b]
Norflurazon	15.224	48.3 [a]	51.8 [b]	4.80 [a]	10.5 [b]	18.2 [a]	99.5 [b]	15.9 [a]	51.4 [b]
Lenacil	15.362	31.9 [a]	77.6 [b]	5.40 [a]	−14.7 [b]	29.4 [a]	99.5 [b]	28.4 [a]	54.7 [b]
Endosulfansulfate	15.36	23.4 [a]	35.9 [b]	4.80 [a]	10.5 [b]	8.00 [a]	19.3 [b]	19.9 [a]	28.9 [b]
p,p'-DDT	15.426	33.2 [a]	43.6 [b]	7.60 [a]	13.8 [b]	63.3 [a]	70.8 [b]	16.9 [a]	28.7 [b]
Hexazinone	15.505	45.3 [a]	60.1 [b]	77.5 [a]	85.0 [b]	25.6 [a]	50.0 [b]	17.4 [a]	19.6 [b]
2,4′-Methoxychlor	15.52	46.4 [a]	52.1 [b]	58.9 [a]	59.6 [a]	8.20 [a]	21.1 [b]	18.7 [a]	26.8 [b]
Methoxychlor	15.679	19.9 [a]	25.4 [b]	22.5 [a]	33.9 [b]	22.9 [a]	40.0 [b]	27.8 [a]	39.8 [b]
Tebuconazole	15.715	57.9 [a]	73.6 [b]	28.7 [a]	37.5 [b]	11.2 [a]	54.9 [b]	18.9 [a]	24.1 [b]

Table 2 (*continued*)

Compound Name	Rt (min)	Okra		Spinach		Pomegranate		Tea	
		JAC	GCB	JAC	GCB	JAC	GCB	JAC	GCB
Propargite-1	15.717	50.7 [a]	53.8 [a]	30.8 [a]	37.8 [b]	15.3 [a]	36.7 [b]	28.6 [a]	32.0 [b]
Propargite-2	15.717	53.2 [a]	61.6 [b]	32.9 [a]	43.5 [b]	24.4 [a]	64.0 [b]	43.7 [a]	54.2 [b]
Piperonylbutoxide	15.809	28.3 [a]	42.6 [b]	83.2 [a]	85.6	19.1 [a]	35.1 [b]	15.3 [a]	19.6 [b]
Nitralin	15.843	67.7 [a]	68.4 [a]	47.7 [a]	59.3 [b]	20.9 [a]	97.0 [b]	24.9 [a]	43.9 [b]
Tetramethrin-1	16.238	39.5 [b]	34.5 [a]	−36.5 [b]	−29.1 [a]	23.6 [a]	29.2 [b]	39.8 [a]	78.9 [b]
Pyridaphenthion	16.175	72.3 [a]	74.3 [a]	−61.1 [a]	−88.7 [b]	25.9 [a]	34.9 [b]	19.5 [a]	23.8 [b]
Bifenthrin	16.33	27.1 [a]	37.4 [b]	−88.3 [b]	−23.7 [a]	13.6 [a]	18.9 [b]	20.0 [a]	27.8 [b]
Phosmet	16.362	70.0 [a]	98.7 [b]	59.6 [a]	62.5 [a]	12.0 [a]	19.0 [b]	16.8 [a]	25.9 [b]
Bromopropylate	16.437	40.4 [a]	52.6 [b]	34.7 [a]	36.4 [a]	11.0 [a]	50.8 [b]	18.6 [a]	19.7 [a]
Fenpropathrin	16.542	29.7 [a]	39.3 [b]	−19.1 [a]	−55.1 [b]	17.3 [a]	20.0 [b]	17.0 [a]	17.9 [a]
Tebufenpyrad	16.666	13.6 [a]	25.7 [b]	52.6 [a]	73.7 [b]	49.8 [a]	70.0 [b]	12.1 [a]	52.2 [b]
cis-Permethrine	16.937	73.5 [a]	79.8 [a]	40.6 [a]	50.5 [b]	24.1 [a]	26.1 [a]	11.5 [a]	26.3 [b]
trans-Permethrine	16.937	18.6 [a]	34.8 [b]	40.2 [a]	46.1 [b]	20.6 [a]	21.6 [a]	6.30 [a]	17.0 [b]
Tetradifon	16.978	33.7 [a]	41.2 [b]	−19.7 [b]	−15.3 [a]	17.3 [a]	26.0 [b]	15.3 [a]	35.4 [b]
lambda-Cyhalothrin	17.36	20.4 [a]	37.1[b]	28.7 [a]	42.5 [b]	8.70 [a]	10.0 [b]	44.0 [a]	52.6 [b]
Mirex	17.548	25.9 [a]	46.3 [b]	8.10 [a]	24.6[b]	16.5[a]	31.0 [b]	11.1 [a]	26.3 [b]
Pyrazophos	17.574	41.3 [a]	41.7 [a]	27.7 [b]	35.5 [b]	31.5 [a]	32.2 [a]	32.9 [a]	36.0 [a]
Fenarimol	17.647	13.6 [a]	19.7 [b]	30.7 [b]	22.5 [a]	21.7 [a]	27.1 [b]	22.8 [a]	27.0 [b]
Azinphos-ethyl	17.747	12.4 [a]	18.4 [b]	17.8	23.3 [b]	15.0 [a]	21.3 [b]	8.10 [a]	24.6 [b]
Etofenprox	17.917	46.9 [a]	54.8 [b]	−19.3 [b]	−11.7 [a]	18.6 [a]	20.3 [a]	22.3 [a]	27.1 [b]
Flucythrinate-1	18.132	67.3 [a]	75.2 [b]	−19.2 [a]	−18.2 [a]	19.7 [a]	23.6 [b]	4.70 [a]	19.7 [b]
Flucythrinate-2	18.132	19.6 [a]	27.5[b]	79.0 [a]	81.9 [a]	20.2 [a]	26.3 [b]	23.1 [a]	28.9 [b]
Acequinocyl deg.	18.158	26.3 [a]	42.2 [b]	17.8 [a]	33.4 [b]	13.4 [a]	14.8 [a]	16.1 [a]	25.6 [b]
Acequinocyl	18.158	16.5 [a]	23.9 [b]	15.2 [a]	31.3 [b]	26.9 [a]	28.5 [b]	8.40 [a]	25.6 [b]
Pyridaben	18.367	30.3 [a]	38.2 [b]	32.4 [a]	35.7 [a]	17.5 [a]	29.4 [b]	16.5 [a]	29.7 [b]
Prochloraz	18.419	27.7 [a]	35.5 [b]	−3.40 [b]	−0.90 [a]	19.2 [a]	50.7 [b]	13.5 [a]	22.6 [b]
Cyfluthrin-1	18.718	39.3 [a]	47.1 [b]	56.1 [a]	61.2 [b]	18.4 [a]	25.4 [b]	11.8 [a]	28.9 [b]
Cyfluthrin-2	18.821	28.7 [a]	36.6 [b]	59.4 [a]	65.5 [a]	17.3 [a]	20.0 [b]	11.5 [a]	26.2 [b]
Cyfluthrin-3	18.885	37.6 [a]	45.4 [b]	37.7 [a]	40.8 [b]	22.1 [a]	27.0 [b]	8.20 [a]	23.1 [b]
Cyfluthrin-4	18.932	22.2 [a]	30.1 [b]	74.9 [a]	77.3 [a]	24.2 [a]	32.2 [b]	18.9 [a]	31.7 [b]
Cypermethrin-1	19.07	44.2 [a]	52.1 [b]	−10.1 [a]	−18.6 [b]	13.9 [a]	18.1 [b]	2.10 [a]	21.4 [b]
Cypermethrin-2	19.186	53.1 [a]	61.0 [b]	83.5 [a]	85.7 [a]	18.0 [a]	34.6 [b]	13.1 [a]	25.3 [b]
Fenvalerate-1	20.176	43.7 [a]	51.5 [b]	57.1 [a]	59.7 [b]	29.5 [a]	52.9 [b]	23.1 [a]	32.6 [b]
tau-Fluvalinate-1	20.318	19.6 [a]	27.5 [b]	62.5 [a]	65.9 [a]	21.0 [a]	24.0 [b]	16.8 [a]	25.0 [b]

Values followed by different letters in superscript within a row are significantly different at 5% level based on Duncan's multiple range test performed using SPSS. Each value represents the mean of six replicates (n = 6).
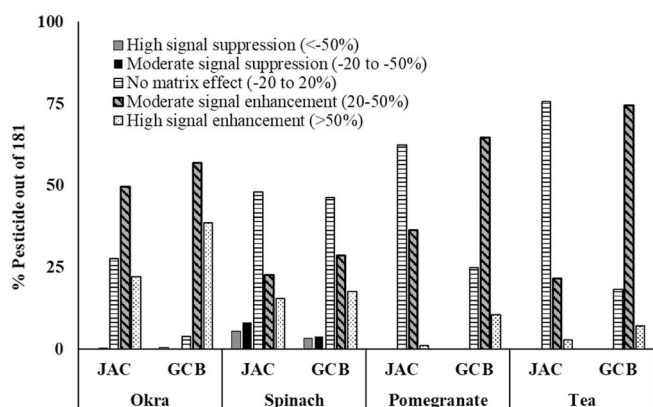

Fig. 5. Matrix effect after the cleanup treatment with JAC and GCB.

Table 3. An overall acceptable recovery was reported in these references, although the dose of the sorbent materials, number of targeted pesticides and matrices had varied. For example, the dosage of GCB varied from 7 to 90 mg per sample of spinach, grape, pomegranate, okra and tomato, where the number of targeted pesticides varied from 10 to 349. The non-GCB adsorbents showed a wide variation in dose (between 0.5 and 100 mg per sample), although their efficiency was evaluated on a relatively less number of pesticides (2–78). When compared, the effectiveness of JAC appeared superior in analysing residues of 181 pesticides in all the tested matrices. Furthermore, a low application rate (only 5 mg per sample) was quite effective in lowering the cost of sample analysis compared to GCB. The information presented in Table 3

indicate that JAC performed better than many of the reported adsorbents with a satisfactory performance across food matrices. Based on the comparative evaluation, JAC's futuristic application in routine pesticide residue testing is well-anticipated.

3.4. Cost analysis of JAC production

With regard to adsorbents (*e.g.* petroleum based GCB, silica based products and carbon nano tubes), JAC can be easily produced from jute stick, which is an underutilised waste available at a very cheap price. The raw material of JAC is a low cost (maximum 0.07 USD per kilogram) bioresource and the tentative cost of JAC (laboratory sacle production) is around USD 10 per kg, *i.e.* USD 0.01 per gram. As opposed, the commercial GCB costs approximately 11–12 USD per gram. Hence, GCB is nearly 1100–1200 folds costlier than JAC. From the above, it is evident that the application of JAC in place of GCB will unquestionably reduce the cost of sample analysis with an overall superior performance in multiresidue cleanup.

4. Conclusion

This study presented how JAC from jute stick was synthesised and characterised by CHNS, SEM, TGA, XRD, FTIR and BET analysis and further investigated the effectiveness of JAC for the cleanup of four complex food matrices in determining pesticide residues. The findings demonstrated JAC as an efficient cleanup agent which satisfactorily removed the matrix co-extractives without affecting the recovery of the targeted pesticide residues. A comparative analysis with GCB indicated a superior performance of JAC in terms of the rate of use, recovery, pesticide chemical-class and its applicability in all chosen matrices. As

Table 3
Comparison with other adsorbents as reported in literature.

Carbonaceous adsorbent	Dose	Total pesticides	Matrices	Recovery (%)	References
GCB based adsorbents					
GCB	10 mg	66	Spinach	66–104	Li et al. (2009)
GCB	7 mg	349	Grape, pomegranate, okra, tomato, onion	70–110	Banerjee et al. (2012)
GCB	75 mg	49	Tobacco	70–119	Khan et al. (2014)
GCB	30 mg	133	Chenpi	70–112.2	Li et al. (2019)
Magnetic GCB	10 mg	10	Cucumber, gourds, cabbage, tomato	69.9–125	Zheng et al., 2013
High surface GCB	90 mg	46	Spinach	74–116	Islam et al. (2019)
Non-GCB adsorbents					
Modified silica nano particle	20 mg	4	Rice, corn, soybean	73.8–110.8	Peng et al. (2010)
Multiwall carbon nanotube (MWCNT)	10 mg	30	Cabbage, spinach, grape, orange	71–110	Zhao et al. (2012)
Amine modified graphene	0.5 mg	28	Rapeseed, peanut, sesame, soybean	70.5–100	Guan et al. (2013)
MWCNT	6 mg	78	Tea	70–120	Hou et al. (2014)
Magnetic- MWCNT	100 mg	8	Tea	72.5–109.1	Deng et al. (2014)
Magnetic graphene	80 mg	26	Tobacco	64–104	Luo et al. (2015)
CNT/β-cyclodextrinnano composite	3.5 mg	2	Tomato	84.2–108.9	Ding et al. (2019)
JAC	5 mg	181	Okra, pomegranate, spinach, tea	66–120	Present study

the application of JAC resulted in a relatively lower ME for most of the pesticides, the study recommends the food testing laboratories to use this novel cleanup agent as a cost-effective alternative of GCB, supplanting the expensive ones. In other words, the results anticipate a new market demand for JAC, particularly in the food testing industries. It would be further useful to see how the application of JAC as a cleanup agent performs in other matrices.

CRediT authorship contribution statement

Rakesh Kumar Ghosh: Investigation, Methodology, Writing – original draft. **Sujan Majumder:** Investigation, Methodology, Writing – original draft. **Arijita Bhattacharyya:** Investigation, Methodology, Writing – original draft. **Anindita Paul:** Investigation, Validation, Formal analysis. **Zareen Khan:** Investigation, Validation, Data curation. **Deb Prasad Ray:** Investigation, Formal analysis, Writing – review & editing. **Sambhu Nath Chattopadhyay:** Investigation, Formal analysis. **Anita Pardeshi:** Validation, Data curation. **D.B. Shakyawar:** Supervision, Project administration. **Kaushik Banerjee:** Investigation, Supervision, Visualization, Writing – original draft, Writing – review & editing, Project administration.

Declaration of competing interest

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Appendix A. Supplementary data

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