Review Paper

A mini-review of the morphological properties of biosorbents derived from plant leaves

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Abstract
A key feature of biosorption studies is the utilisation of different analytical techniques to evaluate several aspects of the process. Scanning electron microscopy (SEM) is used to study the morphology features of biosorbents. In this mini-review, research efforts by scientists in evaluating the morphological properties of plant leaves biosorbents was discussed. The way in which results have been interpreted and what inferences have been deduced was also discussed. Biosorbents obtained from plant leaves generally possess heterogeneous and irregular surface containing a variety of cavities, holes, voids, cracks, interstices and convolutions. NaOH pre-treatment (also known as mercerisation) helps to remove lignin and oil from the biomass as well as increasing the roughness of the base cellulose. Smoother surfaces with fewer features are observed after sorption of some chemical species (be it pollutants or a modification agent). It was observed that impregnation with nanoparticles usually leads to nanoparticle macro-clusters observable at high resolutions while any previously observed cavities, holes, ruptures and voids tends to disappear. Carbonisation and calcination gives a more porous adsorbent. Chemical modification despite reducing the total surface area of the adsorbent can actually increase the adsorption capacity due to adjustments in functional groups, effects on the solution chemistry and the improvement of the inherent affinity of the biosorbent due to the modification. SEM analysis is even more important in recent times where the functionalisation of adsorbents and biosorbents is a common practice.

Keywords Adsorption · SEM · Plant leaves · Biosorbent · Morphology analysis

1 Introduction

Energy and environmental sustainability is of major concern to environmental engineers in contemporary times. In light of these, researchers have over the years developed numerous techniques for the treatment of polluted effluents [1, 2]. A popular technique is adsorption by biomaterials also known as biosorption [3]. A plethora of biological materials have been studied over the years and these includes fish scales [4, 5], plant leaves [6, 7], tree barks [8], agricultural wastes [9], egg shells [10, 11], fruit pods, fruit shells [12] and a host of others. Plant leaves have had a wave of research papers (in biosorption studies) in recent times due to their low cost and availability all year round. Adeniyi and Ighalo [6] observed that nations like India, Pakistan, Malaysia, Turkey and China has a large number of plant leave biosorption studies due to their lingering issues of water pollution, urbanisation and industrialisation (leading to poor management of industrial effluents). In a bid to further assist researchers in their quest for environmental sustainability via plant leave biosorption, there is a need to delve into the analytical findings of the adsorbents themselves.

A key feature of biosorption studies is the utilisation of different analytical techniques to evaluate several aspects of the biosorbent in other to gain an

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understanding of the potentials and suitability of the material to do the intended job. Branueur–Emmet–Teller (BET) analysis is used to study the particle and pore dimensional characteristics of the biosorbents. Fourier transform infra-red spectroscopy (FTIR) done to determine the functional groups and complexes present in the biosorbent that could be responsible for pollutant uptake. X-ray fluorescence (XRF) is used to analyse the major and trace elements in the biomaterials by studying the behaviour of atoms when they interact with radiation. X-ray diffractometry (XRD) to used investigate the properties of the adsorbent as it relates to the crystallinity of the material.

Microscopy is used to study the morphological features materials. We have Optical Microscopy (OM) and Electron Microscopy (SEM). OM produce coloured images but is greatly limited in terms of resolution [13]. Despite the grey-scale images of SEM, resolution 1000 times greater than that of OM can be achieved. Scanning electron microscopy (as well as the transmission electron microscopy) has been used quite a while is studying the surface morphology of adsorbent materials. Transmission Electron Microscopy (TEM) is also used to study particle morphology albeit to give more detailed images at higher resolutions and confirm the presence of specific molecules on the particle surface [14]. The use of TEM is not as common as SEM in open literature probably due to its high cost.

In a recent paper, we discussed the empirical findings of researchers in plant leaves biosorption and evaluated possible knowledge gaps and research trends [6]. In this mini-review, we put forward a discussion of research efforts by scientists in evaluating the morphological properties of plant leaves biosorbents and conclude on how these results have been interpreted and what inferences have been deduced. The possible effects of modification and pollutant loading on the biosorbents are also discussed. The pool of literature is gotten from (but not exclusive to) all studies investigating plant leaves biosorbents using SEM over the past 10 years and obtained from google scholar. Within the scope of the author's exhaustive search, there is no such literature in this regard that carefully examines the morphological properties and transformations of biosorbents from plant leaves. This leaves an important knowledge gap to be filled. A review such as this will serve as an important template to researchers in discussing the images from their SEM analyses, help them gain better understanding of the significance of what they actually see in those micrographs and assist in gaining a deeper understanding of the possible information that can be drawn from these images. 'Biosorbents' in the context of this paper refers to special type of adsorbents that have not undergone any high temperature process (e.g. carbonisation and pyrolysis) and are simply grinded from the source leaves and chemically modified (in some cases) at ambient or near-ambient conditions.

2 Overview of scanning electron microscopy

In this section we take a brief historical examination of SEM analysis to understand the fundamental principles behind the technology and gain a historical preview of its development. In SEM, a fine probe of energised electrons (having energies up to 40 keV) is projected on a specific sample and scanned along a pattern of parallel lines [15]. Various signals are generated due to the incidence of the electrons on the sample surface and these are collected to form an image [15]. These incident rays are mostly secondary electrons (the high energy electrons backscattered from the primary beam). More details of the physics of the process is given by Klein et al. [16]. SEM technology is based on two fundamental discoveries. The first is the observation that trajectories of charged particles in axially symmetric electric and magnetic fields could act as particle lenses indirectly laying the foundations of geometrical electron optics in 1926 [15]. The second was the introduction of the wave electron optics which was based on the concept of the corpuscule (leading to the assigning of a frequency and a wavelength to a charged particle) around the same time. The first commercially available SEM (then known as 'stereoscan') was developed by Cambridge instruments and made available in the open market in 1965 [17]. A more detailed discussion of the background history of SEM development is presented by McMullan [18] and Haguenau et al. [19]. Most SEM now come with dispersive and non-dispersive X-ray analysers as attachments [17]. X-ray dispersive analysis (EDX) works hand in hand with SEM to provide a qualitative and quantitative information about the composition of the material [13]. SEM though initially designed for applications in biology for the examination of inorganic materials [20] has now found applications in many other research areas (such as the one considered in the mini-review)

3 Instrumentation of scanning electron microscopy

A variety of scanning electron microscopes are used in studies and reported in open literature. Procedural overview of SEM analyses is explained in literature [21, 22]. However, most studies on plant leaves biosorption did not give any discussion of the procedure. In the description by Adeniyi et al. [21], a double adhesive was placed on a sample stub. The sample was the sprinkled on the stub and subsequently taken to a sputter coater with

5 nm of gold. The sample was placed on a charge reduction sample holder and introduced into the column of the SEM machine. It was firstly viewed from a Navigation Camera before being sent to SEM mode. The spectrum was recorded using transmittance method in the 4000–650 cm^{-1} region with numerous sample scans. The acceleration voltage of the microscope was set to 15 kV and magnification of 500–1500 times. Different magnifications were stored in a USB stick after adjustment of brightness and contrast. Different SEM models have different image magnification and clarity that is why it is pertinent that the model be stated. It is also to ensure reproducibility of the results. Also, a few studies did not mention the model of SEM machine used in their study [23, 24].

4 Evaluation of analytical findings

4.1 Guava (*Psidium guajava*)

Abdelwahab et al. [24] studied the morphology of unmodified and modified guava leaves biosorbent. The SEM image of the unmodified biosorbent is shown in Fig. 1a while Fig. 1b reveals that of the modified biosorbent. The biosorbent was modified by soaking in a 1 M NaOH solution for 24 h. It was observed that the unmodified biosorbents possess a heterogeneous surface with deep ridged cavities. Upon chemical modification, the surface of the biosorbents became more ridged. Further explanations and elaborations were not given from their observations. Conventionally, NaOH treatment (also known as mercerisation) helps to remove lignin and oil from the biomass as well as increasing the roughness of the cellulose [25]. It was proposed that the adsorption mechanism was either boundary layer diffusion or intra-particle diffusion or both. The findings revealed that both biosorbents adsorbed Cd(II) according to second order kinetics, but clear distinctions between both types was unfortunately not made.

4.2 Aloe Vera

Abedi et al. [26] studied the morphological features unmodified (Fig. 2a) and magnetically modified (Fig. 2b) aloe vera ash biosorbents. The biosorbents were modified using iron oxide nanoparticles and used for the adsorption of Pb(II), Cu(II), Zn(II) and Cr(II). The unmodified biosorbents was observed to have a rough surface structure with flaky micro-particles of which the authors inferred to mean the possible presence of cavities. However, impregnation with iron oxide nanoparticles completely transformed the micro-structure of the adsorbent (see Fig. 2b). The spherical cage-like structures formed was an indication of homogeneity between biosorbent and the nanoparticles. Much

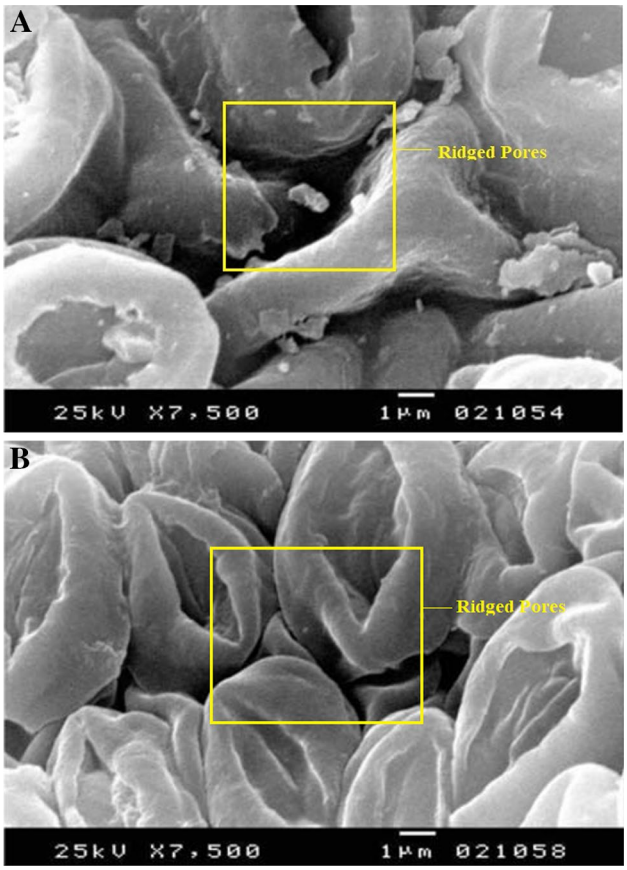

Fig. 1 **a** SEM image of unmodified Guava leaves biosorbent at ×7500 magnification [24]. **b** SEM image of modified Guava leaves biosorbent at ×7500 magnification [24]

of the active sites were observed to have been occupied by the nanoparticles hence the seemingly more homogeneous appearance. For the magnetically modified biosorbent after adsorption of heavy metals, the almost homogenous surface suggests that virtually all cavities on the surface have been occupied by the nanoparticles or pollutants. Overall, it was observed that the heavy metal adsorption process for was rapid and > 98% removal efficiency was achieved at optimised conditions. The morphological observations of Abedi et al. [26] is corroborated by Malik et al. [27] in their study.

4.3 Tea

The morphology of spent black tea leaves before and after phenol adsorption was evaluated in a study by Ali et al. [28]. The original adsorbent (Fig. 3a) had a diversified, coarse and highly porous surface composed of numerous fibrous bonds and spongy formations. After the adsorption of phenol (Fig. 3b), much of the observed cavities were no longer observed and the surface had a more homogenous appearance. This is quite similar to the

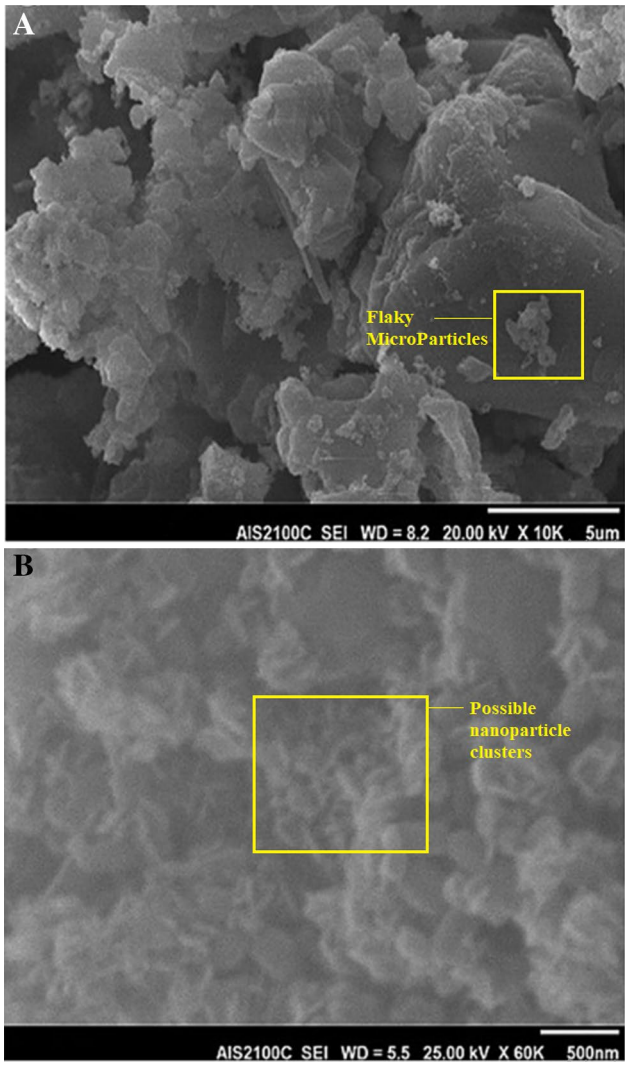

Fig. 2 **a** SEM image of unmodified Aloe vera leaves ash biosorbent at ×10,000 magnification [26]. **b** SEM image of magnetically modified Aloe vera leaves ash biosorbent at ×60,000 magnification [26]

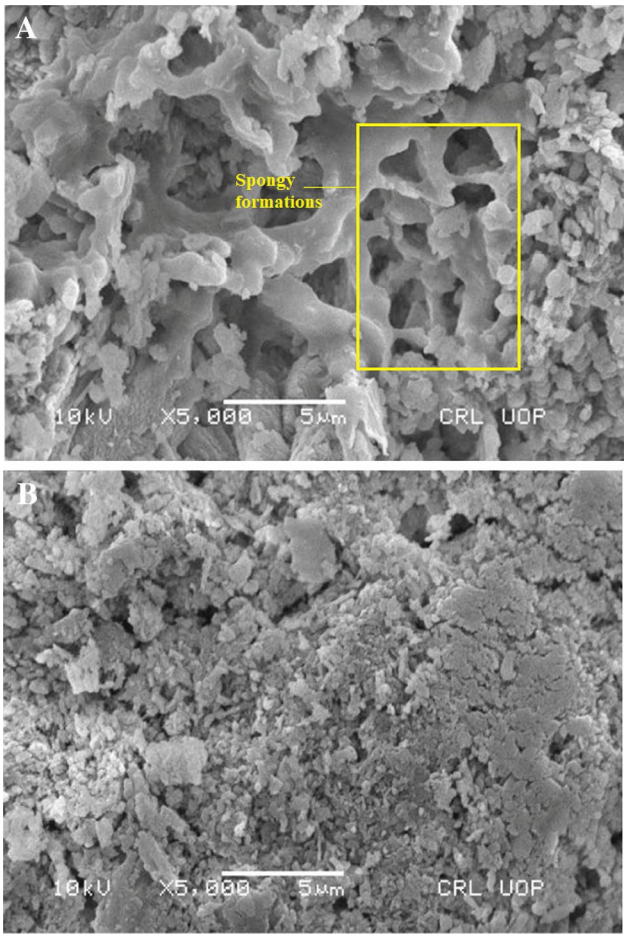

Fig. 3 **a** SEM image of spent black tea leaves at ×5000 magnification [28]. **b** SEM image of spent black tea leaves after adsorption at ×5000 magnification [28]

observations of Abedi et al. [26] albeit for aloe vera. Bajpai and Jain [29] also examined spent tea leaves at a lower magnification of 50× and 800× (images not shown). Their study was however focused on the adsorption of crystal violet. The macro-structure of the biosorbent particles could be clearly noticed from the images and the surface heterogeneity summarily confirmed.

4.4 Neem (*Azadirachta indica*)

The SEM image of Neem leaves powder by Bharali and Bhattacharyya [23] revealed a surface with a porous look with a large number of craters (Fig. 4a). Several irregular convolutions also seem to appear on the surface. After sorption of fluoride (Fig. 4b), the porous outlook was partly destroyed as the craters were no longer noticeable. The authors attributed this to the sorption of fluoride unto the cavities and over the cracks on the surface of the biosorbent. Neem leaves was found to be an effective adsorbent in the uptake of fluoride from aqueous solution (> 80% removal efficiency). The morphological findings of Bharali and Bhattacharyya [23] is in consonance with the previously observed reduction in heterogeneity due to the adsorption of some chemical species. Bhattacharyya and Sharma [30] had earlier examined neem leaves powder albeit at a higher magnification of 4000× and 20,000× (images not shown) in a study focused on Pb(II) biosorption. Similar convolutions, irregularities and broken edges was also observed.

4.5 Bael (*Aegle marmelos*)

Chakravarty et al. [31] examined the morphology of Beal leaves powder adsorbent for loaded (Fig. 5a) and unloaded (Fig. 5b) specimens. The study evaluated the

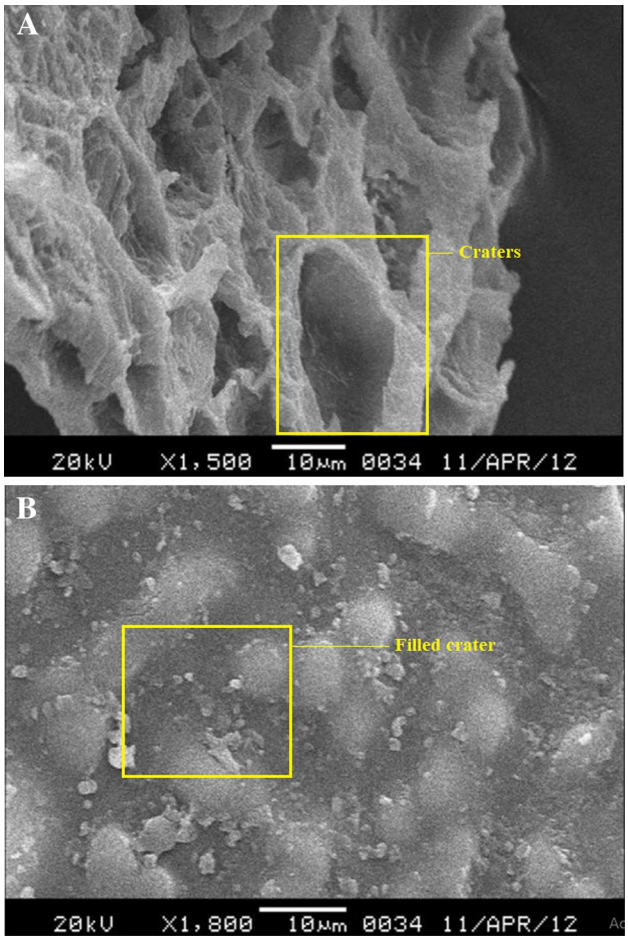

Fig. 4 **a** SEM image of Neem leaves powder at ×1500 magnification [23]. **b** SEM image of Neem leaves powder after adsorption at ×1500 magnification [23]

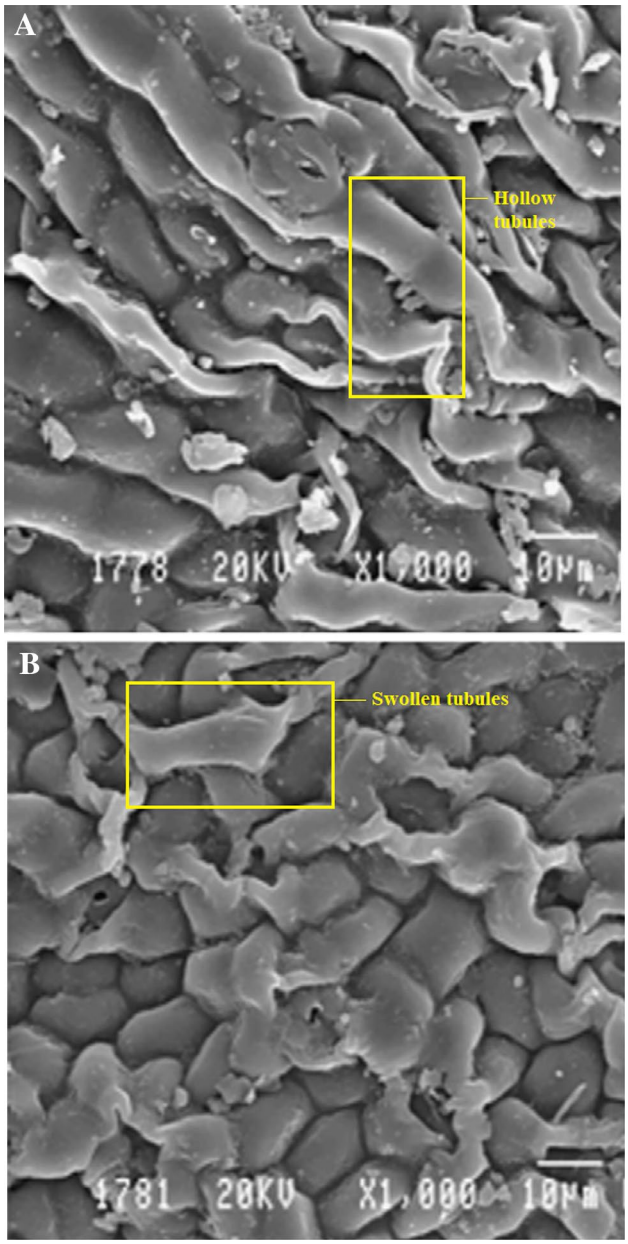

Fig. 5 **a** SEM image of Bael leaves powder at ×1000 magnification [31]. **b** SEM image of Bael leaves powder after adsorption at ×1000 magnification [31]

biosorption of Pb(II). The un-used/unloaded specimens showed a regular symmetry with hollow tubular structures. After biosorption of Pb(II), the tubes become noticeably swollen as the metal enters the fibers of the leaves. It was put forward by the authors that Pb(II) is adsorbed to the functional groups present inside the wall of the tubular structures of the leaves. This inference was corroborated by their FTIR analysis. The biosorption process was described as physisorption and an adsorption capacity of about 104 mg/g was calculated. At a resolution ten times that of Chakravarty et al. [31], Sahu et al. [32] observed the hollow tubular structures of bael leaves biosorbent and some cavities. The study went on to observe the smoothening out of the surface after impregnation with ferromagnetic nanoparticles.

4.6 Coconut (*Cocos nucifera L.*)

Jawad et al. [33] studied the morphology of unused (Fig. 6a) and used (Fig. 6b) adsorbent obtained from coconut leaves. The adsorbent was used for the sorption of methylene blue. From the micrographs, chemical activation with phosphoric acid was useful in developing a mesoporous structure within the carbon. The pores are formed due to the evaporation of the activating agent during high temperature carbonisation. This therefore

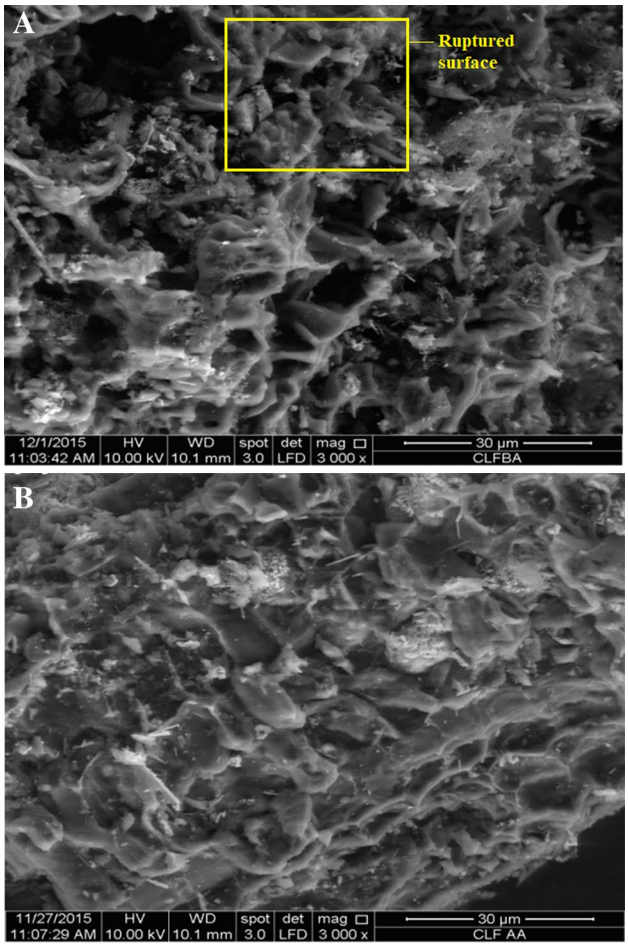

Fig. 6 **a** SEM image of coconut leaves activated carbon at ×3000 magnification [33]. **b** SEM image of coconut leaves activated carbon after adsorption at ×3000 magnification [33]

leaves behind a ruptured surface of the carbon with numerous cavities previously occupied by the activating chemical agent. The surface area of the activated carbon was very high at 981.79 m^2/g. The external surface has cracks and crevices as well as irregular and heterogeneous morphology with a well-developed porous structure in various sizes. Due to the presence of these cavities, it was proposed by the authors the adsorbent will have a better capacity for methylene blue molecules as they can diffuse into these cavities and get trapped on active sites. Figure 6b reveals that the above assumption is valid. The activated carbon surface became denser and with less open cavities on the surface.

4.7 *Tephrosia purpuria*

Madala et al. [34] studied the morphology of *Tephrosia purpuria* leaves biosorbent. The unmodified (Fig. 7a), acid modified (Fig. 7b) and used biosorbent (Fig. 7c) were

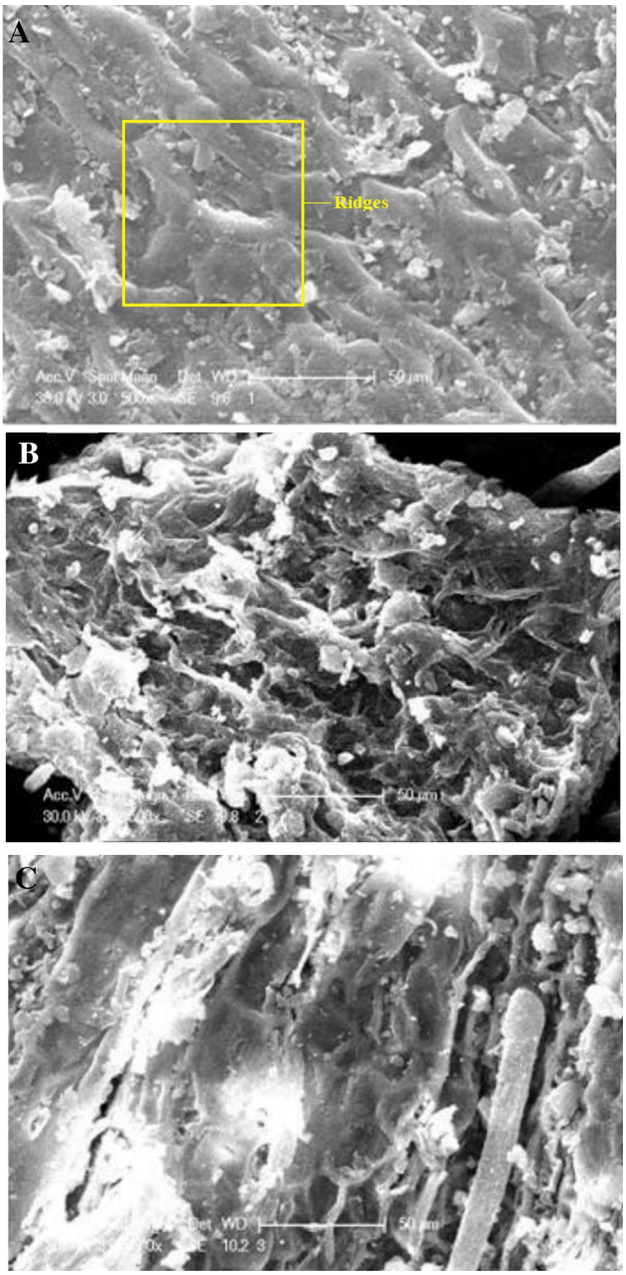

Fig. 7 **a** SEM image of raw *Tephrosia purpuria* leaves biosorbent at ×500 magnification [34]. **b** SEM image of modified *Tephrosia purpuria* leaves biosorbent at ×500 magnification [34]. **c** SEM image of modified *Tephrosia purpuria* leaves biosorbent after adsorption at ×500 magnification [34]

examined. The unmodified biosorbent had a uniform and regular structure with observable ridges. After acid modification, the surface of the biosorbent became more porous and heterogeneous due to ruptures induced by the acid treatment. Akhabue et al. [35] observed that acid treatment of biomass leads to ruptures on the surfaces. After adsorption, there was a noticeably smoothening out of the biosorbent surface as Pb(II) ions are adsorbed onto

the adsorbent. The modified biosorbent had an adsorption capacity of 100 mg/g and the process was highly feasible, spontaneous and exothermic.

4.8 Rubber (*Hevea brasiliensis*)

Fadzil et al. [36] described modified (Fig. 8b) and unmodified (Fig. 8a) rubber leaves biosorbent in their study. The biosorbent was modified with monosodium glutamate. Both the unmodified and modified biosorbents were described as jagged, irregular in shape and with nooks and crannies. Though modification did not make the surface smoother, the modified biosorbent had a less jagged look but with identifiably organised crevices running on the surface (Fig. 8b). A similar observation was made by Kamal et al. [37] for chemical treated rubber leaves, though the irregularities on the surface of the biosorbent was greater in their case. Nag et al. [38] was also able to show that rubber leaves biosorbents will reduce in surface heterogeneity after pollutant sorption. Other researchers have also examined the micrographs of rubber leaves biosorbents [39, 40].

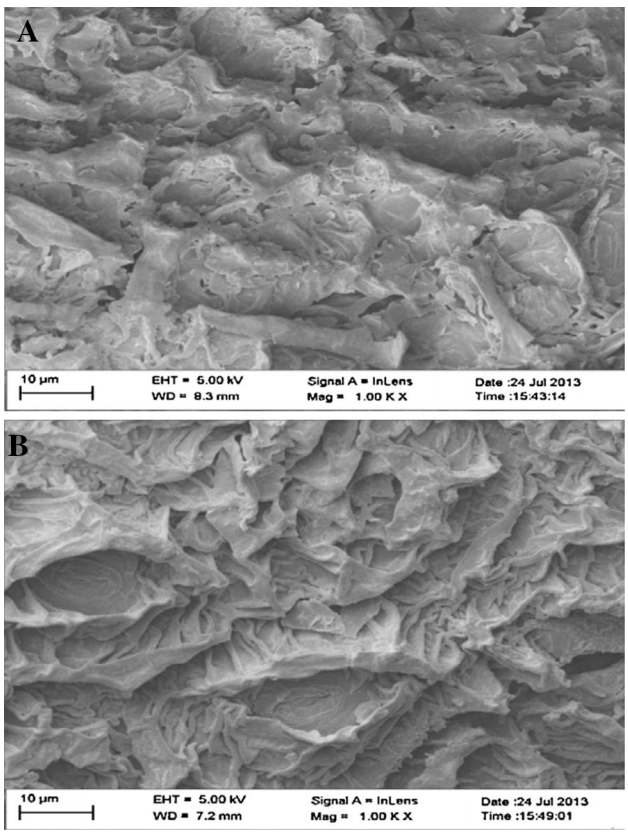

Fig. 8 **a** SEM image of Rubber leaves biosorbent at ×1000 magnification [36]. **b** SEM image of modified Rubber leaves biosorbent at ×1000 magnification [36]

5 Comparison of adsorption capacities of plant leaves derived biosorbents

In this section, a comparison is made of the adsorption capacities of raw and modified biosorbents derived from plant leaves for different pollutants. The comparison for heavy metals is shown in Table 1 and for other pollutant in Table 2. The discussion in this section was restricted to studies where direct comparison was made in this domain. It would be erroneous to make generalisations across several studies as not only chemical modification can affect the adsorption capacity. Other factors like functional groups, solution chemistry, pH, sorbate-sorbent interphase and affinity can also affect the adsorption capacity. Only a few studies directly compared the modified and unmodified adsorbents. Dabbagh et al. [41] compared the adsorption capacities of *Ficus carica* for Co(II) for unmodified adsorbent and those modified with HCl and $MgCl_2$. At a pH of 6, the adsorption capacity of the $MgCl_2$ modified adsorbent was 33.90 mg/g followed by the untreated (14.80 mg/g) and then the HCl modified (9.20 mg/g). The biosorbent adsorption capacity can be improved for heavy metal sorption by reducing the electronegativity of the biomass surface. However, Dabbagh et al. [41] explained that acid treatment could cause a reduction in this electronegativity due to the protonation by the acid. The Mg^{2+} ions however are will readily exchange with the Co(II) thereby making the biosorbent to serve as a kind of ion-exchange resin. The morphological changes were unreported. El-Sayed and Nada [14] observed that oil palm leaves modified by polyethylenimine has a lesser specific surface area but a higher pollutant removal efficiency for Cr(VI) and Pb(II). Despite the reduction in the surface area, the polyethylenimine helped to functionalise the biosorbent and improve its affinity for the heavy metals.

Fadzil et al. [36] compared the use of citric acid and monosodium glutamate (MSG) in the modification of rubber leaves for Pb(II) adsorption. They observed that MSG was the better modification agent albeit without any comparison made to the unmodified leaves. Mondal et al. [42] utilised anionic surfactant sodium dodecyl sulphate (SDS) and non-ionic surfactant triton X-100 to modify bamboo leaf powder for the adsorption of mercury. The unmodified powders had a lesser adsorption capacity for mercury (27.11 mg/g) than the SDS (31.05 mg/g) and triton modified (27.10 mg/g) biosorbents. It should be noted that washing with NaOH is not considered a modification technique. It is done to leach out solubles like tannin, chlorophyll and lignin from the plant materials [6]. This is a prerequisite for it to be suitable used as an adsorbent (except when extensive

Table 1 Adsorption capacity of plant leave biosorbents for heavy metals

Reference	Leaves	Metal	pH	Adsorption capacity before modification (mg/g)	Adsorption capacity after modification (mg/g)	Modification agent
Abedi et al. [26]	Aloe Vera ash	Pb^{2+}	6	–	333.3	Fe_3O_4
		Cu^{2+}	6	–	3445	Fe_3O_4
		Zn^{2+}	6	–	71.40	Fe_3O_4
		Cr^{3+}	6	–	333.3	Fe_3O_4
Aditya et al. [43]	*E. variegata orientalis*	Cr^{6+}	3	6.320	–	–
Ahluwalia and Goyal [44]	Tea	Pb^{2+}	5	2.096	–	–
		Fe^{3+}	5	79.53	–	–
		Zn^{2+}	5	785.6	–	–
		Ni^{2+}	5	515.1	–	–
Al Rmalli et al. [45]	Castor	Hg^{2+}	5.5	37.20	–	–
Ananthakumar et al. [46]	Neem	Cr^{6+}	5	–	6.925	H_2SO_4
Aoyama et al. [47]	Black locust	Cr^{6+}	2	–	25.75	HNO_3
Aoyama [48]	London Plane	Cr^{6+}	3	–	83.33	HNO_3
Babu and Gupta [49]	Neem	Cr^{6+}	2	–	62.97	HCl
Bhattacharyya and Sharma [30]	Neem	Pb^{2+}	7	300.1	–	–
Çekim et al. [50]	Tobacco	Cu^{2+}	4	–	17.18	H_2SO_4
Chakravarty et al. [31]	Bael	Pb^{2+}	5.1	104.0	–	–
Chen et al. [51]	*C. camphora*	Pb^{2+}	5	75.82	–	–
Abdel-Ghani et al. [52]	*T. domingensis*	Al^{3+}	2.5	0.348	–	–
		Fe^{3+}	2.5	2.114	–	–
		Zn^{2+}	2.5	0.201	–	–
		Pb^{2+}	2.5	0.654	–	–
Cheraghi et al. [53]	Sesame	Cd^{2+}	5.5	84.74	–	–
Choudhary et al. [54]	*L. speciosa*	Pd^{2+}	2	–	56.50	H_2SO_4
Dabbagh et al. [41]	*F. carica*	Co^{2+}	6	33.90	14.80	$MgCl_2$
Edokpayi et al. [22]	*D. eriocarpum*	Pb^{2+}	4	41.49	–	–
El-Sayed and Nada [14]	Oil palm	Cr^{6+}	2	–	125.0	Polyethylenimine
		Pb^{2+}	6	–	142.9	Polyethylenimine
Eid [55]	Oil palm	Hg^{2+}	5	–	71.40	Fe_3O_4 and Polyethylenimine
Esheewe et al. [56]	Tea	Pb^{2+}	4	192.3	–	–
Fadzil et al. [36]	Rubber	Pb^{2+}	5	–	97.19	Citric acid
	Rubber	Pb^{2+}	5	–	109.9	Monosodium glutamate
Ghosh et al. [57]	Tea	Cu^{2+}	5.5	7.813	–	–
Gutha et al. [58]	Tomato	Ni^{2+}	5.5	58.82	–	–
Hanafiah et al. [59]	Rubber	Cd^{2+}	5	3.680	–	–
Hanafiah et al. [60]	*I. cylindrica*	Cu^{2+}	5	11.64	–	–
Hossain et al. [61]	Maple	Pb^{2+}	6	125.9	–	–
Hymavathi and Prabhakar [62]	Coconut	Co^{2+}	5	3.691	–	–
Ince et al. [63]	Tea	Cu^{2+}	5	3.360	–	–
Jorgetto et al. [64]	Brazilian orchid	Cu^{2+}	5	15.12	–	–
		Cd^{2+}	5	12.70	–	–
Kaliannan et al. [65]	*S. officinarum* AC	Pb^{2+}	7	148.0	–	–
		Zn^{2+}	7	137.0	–	–
Kamal et al. [37]	Rubber	Pb^{2+}	4	–	95.30	$KMnO_4$ and Na_2CO_3

Table 1 (continued)

Reference	Leaves	Metal	pH	Adsorption capacity before modification (mg/g)	Adsorption capacity after modification (mg/g)	Modification agent
Kamar et al. [66]	Cabbage	Pb^{2+}	6	6.310	–	–
		Cu^{2+}	6	5.740	–	–
		Cd^{2+}	6	5.070	–	–
Kamsonlian et al. [67]	Mango	As^{3+}	7	214.4	–	–
King et al. [68]	*S. cumini* L.	Pb^{2+}	6	32.47	–	–
Kumar et al. [69]	Teak	Cu^{2+}	5	15.43	–	–
Kumar et al. [70]	Teak	Zn^{2+}	5	16.42	–	–
Kumar and Gayathri [71]	Bael	Pb^{2+}	5	4.065	–	–
Kumar and Kirthika [72]	Bael	Ni^{2+}	6.2	1.527	–	–
Kuppusamy et al. [73]	Green honey myrtle	Cr^{6+}	7	62.50	–	–
Li et al. [74]	*I. cylindrica*	Ni^{2+}	7	–	19.14	H_2SO_4
Li et al. [74]	*I. cylindrica*	Ni^{2+}	7	–	19.28	H_3PO_4
Liang et al. [75]	Phoenix	Pb^{2+}	5	71.00	–	–
Madala et al. [34]	*T. purpuria*	Pb^{2+}	6	–	100.0	HNO_3
Khokhar and Siddique [76]	*M. azedarach* L.	Pb^{2+}	–	35.06	28.50	HCl
		Fe^{3+}	–	38.46	28.57	HCl
Martins et al. [77]	Castor	Cd^{2+}	5	38.22	–	–
		Pb^{2+}	5	67.75	–	–
Mohammed et al. [78]	Black tea	Pb^{2+}	5	19.70	–	–
Mondal [79]	Tea	Pb^{2+}	5.8	1.351	–	–
Mondal et al. [42]	Bamboo	Hg^{2+}	8	27.11	31.05	Sodium dodecyl sulphate
Mondal et al. [42]	Bamboo	Hg^{2+}	8	27.11	28.10	Triton
Mozumder et al. [80]	Tea	Cr^{6+}	2	73.10	–	–
Nag et al. [38]	Rubber	Cr^{6+}	1.5	22.97	–	–
Nagpal et al. [81]	Paper mulberry	Cu^{2+}	5.5	43.40	–	–
		Pb^{2+}	5.5	43.90	–	–
		Cd^{2+}	5.5	30.70	–	–
Nakkeeran et al. [82]	*C. esculenta*	Cr^{6+}	2	47.62	–	–
Ngah and Hanafiah [39]	Rubber	Cu^{2+}	4	8.920		
Ngah and Hanafiah [40]	Rubber	Cu^{2+}	4	–	8.710	HCHO and H_2SO_4
Pandey et al. [83]	Kush grass	Cd^{2+}	6.5	19.84	–	–
	Bamboo	Cd^{2+}	6.5	19.71	–	–
Qaiser et al. [84]	*F. religiosa*	Pb^{2+}	4	–	37.45	HNO_3
Rafatullah et al. [85]	Oil palm	Cu^{2+}	6	–	86.95	Sodium dodecyl benzene sulfonate
Rangabhashiyam et al. [86]	*F. auriculata*	Cr^{6+}	2	9.803	–	–
Rao et al. [87]	*F. religiosa*	Cd^{2+}	5.5	27.14	–	–
Reddy et al. [88]	Moringa	Pb^{2+}	5	209.5		
Reddy et al. [89]	Moringa	Cd^{2+}	5	–	171.4	Citric acid
		Cu^{2+}	5	–	167.9	Citric acid
		Ni^{2+}	5	–	163.9	Citric acid
Sahu et al. [32]	Bael	As^{5+}	3	–	69.65	Fe_2O_3

Table 1 (continued)

Reference	Leaves	Metal	pH	Adsorption capacity before modification (mg/g)	Adsorption capacity after modification (mg/g)	Modification agent
Sangi et al. [90]	*U. carpinifolia*	Pb^{2+}	5	201.0	–	–
		Cd^{2+}	5	80.00	–	–
		Cu^{2+}	5	69.50	–	–
	F. excelsior	Pb^{2+}	5	172.0	–	–
		Cd^{2+}	5	67.17	–	–
		Cu^{2+}	5	33.14	–	–
Sert et al. [91]	*P. orientalis*	La^{2+}	4	28.65	–	–
		Ce^{2+}	4	32.05	–	–
Sharma and Bhattacharyya [92]	Neem	Cr^{6+}	5.5	145.8	–	–
Sharma and Bhattacharyya [93]	Neem	Cd^{2+}	9.5	250.0	–	–
Shi et al. [94]	Arborvitae	Pb^{2+}	5.5	35.84	–	–
		Cu^{2+}	5.5	7.940	–	–
		Co^{2+}	5.5	6.780	–	–
Van Suc and Son [95]	Mistletoe	Pb^{2+}	5.5	68.53	–	–
		Cd^{2+}	5.5	50.07	–	–
Venkateswarlu et al. [96]	Neem	Cr^{6+}	7	49.15	–	–
Venkateswarlu et al. [97]	*E. variegata orientalis*	Zn^{2+}	7	17.24	–	–
Vilvanathan and Shanthakumar [98]	Teak	Ni^{2+}	6	18.10	–	–
		Co^{2+}	5	29.50	–	–
Weng and Wu [99]	Pineapple	Cu^{2+}	5	9.280	–	–
Yuvaraja et al. [100]	*S. melongena*	Pb^{2+}	5	71.42	–	–
Zolgharnein et al. [101]	*C. speciosa*	Pb^{2+}	5	120.2	–	–

washing is done). The comparison of adsorption capacity of plant leaves for heavy metals and other pollutants are summarised in Tables 1 and 2 respectively.

6 Appraisal of other recent literature

In this section, we will give a cursory discussion (but without images) of other plant leaves biosorption studies that conducted SEM analysis on their biosorbent. The analysis of Cheraghi et al. [53] on sesame (*Sesamum indicum*) leaves revealed that the biosorbent had a rough and irregular surface. This of course would mean a large area for interaction between the pollutants ions and the adsorbent surface. After the biosorption of Cd(II), a smoothening effect was perceived as the surface became more homogenous. The interstitial spaces, cavities and channels as also observed to have been smoothened out. Cunha et al. [119] studied the biomass precursor and the prepared adsorbents from *Eragrostis plana* leaves. The images revealed that the surface texture of the biomass are quite different. The biomass looked like a dense and low rugosity material without cavities. The prepared biosorbent showed an irregular surface These channels present on the surface of activated carbon allow the passage of the liquid solution through the carbon surface, allowing the mass transfer of the adsorbate from the bulk solution to the surface of the activated carbon.

The analysis of *Diceriocaryum eriocarpum* leave biosorbent by Edokpayi et al. [22] revealed a fluffy and highly porous and rough microstructure containing some voids and cracks. The authors' concluded that the biosorbent is suitable for the adsorption of Pb(II). Gupta et al. [112] described ashoka (*Saraca asoca*) leaves biosorbent as rough, irregular and laden with cavities. Han et al. [113] described poplar (*Populus tremula*) leaves as rough, highly heterogeneous, rough and with a porous structure. The image of the biosorbent after adsorption of methylene blue was significantly different but not necessarily smoother. The authors attributed the difference to an effective adsorption of the pollutants. They furthermore modified sulphuric acid and phosphoric to get more porous products. Hossain et al. [61] described maple (*Acer spp.*) leaves biosorbent as possessing asymmetric cavities

Table 2 Adsorption capacity of plant leave biosorbents for other pollutants

Reference	Leaves	Pollutant	pH	Maximum adsorption capacity before modification (mg/g)	Maximum adsorption capacity after modification (mg/g)	Modification agent
Agarry et al. [102]	Tea	Naphthalene	7	23.81	–	–
Tanim-al-Hasan et al. [103]	Black tea	Basic violet 3	6	372.2	–	–
Bajpai and Jain [29]	Tea	Crystal violet	8	285.7	–	–
Bharali and Bhattacharyya [23]	Neem	Fluoride	6.8	9.500	–	–
Bhattacharyya and Sarma [104]	Neem	Brilliant green	6.5	253.5	–	–
Bhattacharyya and Sharma [105]	Neem	Congo red	6.7	128.3	–	–
Bhattacharyya and Sharma [106]	Neem	Methylene Blue	–	19.61	–	–
Chowdhury et al. [107]	Pineapple	Basic green 4	9	54.64	–	–
Deniz and Saygideger [108]	*P. tomentosa* Steud.	Acid Orange 52	2	10.50	–	–
Dülger et al. [109]	Sumac	Methylene blue	7	151.7	–	–
Elmorsi [110]	Miswak	Methylene blue	10.6	200.0	–	–
Gaikwad and Kinldy [111]	Guava	Auramine	9	7.760	–	–
Gupta et al. [112]	*Saraca asoca*	Methylene blue	6	90.90	–	–
		Malachite green	6	83.30	–	–
		Rhodamine B	6	66.60	–	–
		Brilliant green	6	125.0	–	–
Han et al. [113]	Poplar	Methylene Blue	9	135.4	–	–
Khan et al. [114]	Mango	Rhodamine B	6	3.310	–	–
Liu et al. [115]	Boston Ivy	NH_4^-	5.5	6.590	–	–
Malana et al. [116]	Banyan	Malachite green	–	17.76	–	–
		Methylene blue	–	13.51	–	–
		Methyl orange	–	2.863	–	–
Mandal et al. [117]	Bamboo	Methyl orange	7	125.0	200.0	Sodium dodecyl sulphate
Mandal et al. [117]	Bamboo	Methyl orange	7	125.0	143.0	Triton
Peydayesh and Rahbar-Kelishami [118]	*P. orientalis*	Methylene blue	–	114.9	–	–

and they inferred that this could be the reason for the high surface area of the adsorbent. Hanafiah et al. [60] observed that biosorbents obtained from *Imperata cylindrica* are interestingly non-porous but with an irregular surface. After adsorption, SEM did not observe any major changes in the morphology of the surface of the biosorbents. Li et al. [74] also observed an irregular surface with very few cavities.

Jorgetto et al. [64] described biosorbent particles from Brazilian orchid (*Bauhinia forficata*) leaves as relatively homogenous in size and irregular in shape. However, the microscopic structure is considered to be very rough. The authors furthermore opined that the particles are non-porous based on their observations. The observation of cabbage (*Brassica spp.*) leaves biosorbent by Kamar et al. [66] revealed a highly convoluted surface with heterogeneous crevices. Liang et al. [75] observed that calcination of biosorbents obtained from phoenix tree (*Firmiana simplex*) leaves gave a more porous and rough adsorbent. Makeswari and Santhi [120] studied Castor (*Ricinius communis*) adsorbents using SEM. Their study revealed that activation with $ZnCl_2$ helped to improve the micro-porosity of the adsorbent. This was also observed by Makeswari and Santhi [121].

Scanning electron microscopy (SEM) have been used in numerous other plant leaves biosorption studies.

Chowdhury et al. [107] and Weng et al. [122] examined pineapple (*Ananas comosus*) leaves while Choudhary et al. [54] examined Pride of India (*Lagerstroemia speciose*) leaves. Kaliannan et al. [65] described *Saccharum officinarum* leaves. Kamsonlian et al. [67] and Khan et al. [114] described mango (*Mangifera indica*) leaves biosorbents in their respective studies. Liu et al. [115] studied the morphology of particles obtained from Boston ivy (*Parthenocissus tricuspidata*) leaves. Nagda and Ghole [123] studied tendu (*Diospyros melanoxylon*) leaves biosorbents. Nagpal et al. [81] studied paper mulberry (*Broussonetia papyrifera*) leaves biosorbents. Peydayesh and Rahbar-Kelishami [118] studied Oriental plain (*Platanus orientalis*) leaves powder. Qaiser et al. [84] studied Sacred fig (*Ficus religiosa*) leave powder. Rafatullah et al. [85] and Soliman et al. [124] studied oil palm leaves powder. Rangabhashiyam et al. [86] studied Roxburgh fig (*Ficus auriculata*) leaves powder. Reddy et al. [88] studied *Moringa oleifera* leaves powder. Van Suc and Son [95] studied mistletoe (*Ramus loranthi*) leaves powder. Vilvanathan and Shanthakumar [98] studied teak (*Tectona grandis*) leaves powder. Yuvaraja et al. [100] studied Eggplant (*Solanum melongena*) leaves powder. Most of these studies reinforced the earlier observations from the analytical findings. The forgoing conclusions helps to marry theses inferences together.

7 Knowledge gap

The morphological investigations of biosorbent powders from plant leaves has revealed some very interesting observations over the years. It must be said that a broad spectrum of leaves from various plant species has been examined and the scope of investigation is definitely not lacking in width (but in depth). There are still some research gaps in terms of the depth of investigation. Most studies simply utilise SEM to emphasise the heterogeneity of the adsorbent surface and stop at that. Others proceed further to examine post-adsorption or post-modification transformations. There are a dearth of studies looking at the effect of preparation and/or pretreatment on the final particle morphology. What are the results of the basic physical and chemical procedures and how do they affect morphological transformations from the raw leaves? Furthermore, in studies where desorption has been conducted, there is need to examine the biosorbent morphology after regeneration. Does it remain morphologically similar to the parent adsorbent? If there are changes, does it affect the mechanism of adsorption for subsequent runs? These are few interesting avenues that can be pursued in the domain of the morphology of biosorbents from plant leaves.

8 Conclusion

Biosorbents obtained from plant leaves generally possess highly heterogeneous and irregular surface containing lots of cavities, holes, ruptures, voids, cracks, crevices, nooks, crannies, interstices and convolutions. These features are the sites that help improve surface sorption. The greater the heterogeneity of the surface of the particle, the greater the surface area and the more suitable it is for sorption of pollutants. Smoother surfaces with fewer and less convolutions and other uneven features are observed after sorption of some chemical species or impregnation with a chemical modification agent. Conventionally, NaOH treatment (also known as mercerisation) helps to remove lignin and oil from the biomass as well as increasing the roughness of the base cellulose. Impregnation with nanoparticles usually leads to some specific morphological transformations of the adsorbent. Nanoparticle macro-clusters are usually observable at high resolutions while any previously observed cavities, holes, ruptures and voids tends to disappear. Similar observation is made for biosorbents that have been loaded with heavy metals (after the adsorption process). Carbonisation and calcination gives a more porous and rough adsorbent because volatiles within the microstructure of the particles escape due to high temperature thereby leaving behind voids which end up as pores. It was also observed that chemical modification despite reducing the total surface area of the adsorbent can actually increase the adsorption capacity due to adjustments in functional groups, effects on the solution chemistry (controlled by the pH), sorbate-sorbent interphase and the improvement of the inherent affinity of the biosorbent due to the modification. SEM analysis is even more important in recent times where the functionalisation of adsorbents and biosorbents is a common practice as it can give information on the nature of changes these agents make on the surface morphology.

Data availability Data sharing not applicable to this article as no datasets were generated or analysed during the current study

Compliance with ethical standards

Conflict of interest The authors declare that there are no conflicts of interest.

Human and animal rights This article does not contain any studies involving human or animal subjects.

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