**Walnut Shell based adsorbents: a review study on preparation, mechanism, and application**

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**Supplementary information**

Table S1. Percent content of chemicals in WS reported in Figure 1.

|  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- |
| Ash | Extracts | Cellulose | Hemicellulose | Lignin | Soluble Lignin | Others | Reference |
| 1.1 | 0 | 60.2 | 13.2 | 18.6 | 0 | 6.9 | [1] |
| 0.9 | 4.1 | 26.7 | 23.4 | 49.8 | 0 | 0 | [2] |
| 0 | 0 | 39.6 | 18.5 | 40.6 | 0 | 1.3 | [3] |
| 2.5 | 2.5 | 18.8 | 27.6 | 33.3 | 3.1 | 12.2 | [4] |
| 1.5 | 2.8 | 23.3 | 20.4 | 53.5 | 0 | 0 | [5] |
| 0 | 0 | 25.6 | 22.1 | 52.3 | 0 | 0 | [6] |

Table S2. Summary of the 73 articles used in the review paper.

|  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- |
| Article title | Journal | Target contaminant | Activation method | Adsorption capacity (mg/g) | Annotation | Reference |
| Removal of oil by walnut shell media | Bioresource Technology | oils | None |  |  | [7] |
| Removal of methylene blue from aqueous solutions using agricultural residue walnut shell | Journal of chemistry | Methylene blue | No activation. Pretreatment: rinsed with distilled water and dried in the oven at 105 C | 51.55 |  | [8] |
| Selective adsorption of phenanthrene dissolved in tween 80 solution using AC derived from WS | Chemosphere | Phenanthrene | Washed with distilled water and dried over night at 80 C. First impregnated with 30% phosphoric acid and then thermally activated under nitrogen atmosphere in a furnace at 700 C for 2 hrs | 247.54 |  | [9] |
| Batch adsorption of cephalexin antibiotic from aqueous solution by walnut shell-based activated carbon | Journal of the Taiwan institure of chemical engineers | cephalexin antibiotic | Walnut shell was mixed with ZnCl2 solution at 1–1 impregnation ratio and was stirred for 6 h. The mixture was dried in an oven at 105°C for 24 h. Then, it was placed in a vertical stainless-steel reactor under high-purity nitrogen and flow rate of 300 cm3/min to a final temperature of 450 °C at a heating rate of 5°C/min and was kept at this temperature for 1 h to be activated |  |  | [10] |
| A new absorbent by modifying walnut shell for the removal of anionic dye: Kinetic and thermodynamic studies | Bioresource Technology | anionic dye: C25H14Cl2N7Na3O10S3 | Three step process: 1- washed with distilled water and dried at 105 C for 24 hrs. Then, WS inserted into a mixture of NaOH (1.25 M) and epichlorohydrin at 40 C for 30 mins. Finally, the WS was then added to a mixture of NaOH (0.125 M) and DETA at 65 C and stirred for 1 hr |  |  | [11] |
| Effective modified walnut shel adsorbent: synthesis and adsorption behavior for Pb and Ni from aq solution | Environmental engineering science | Pb and Ni | The adsorbent was prepared by reacting it with maleic acid and N-methyl pyrrolidone for 4 hours in a flask at 90 C |  |  | [12] |
| Application of experimental design methodology to optimize antibiotics removal by walnut shell based activated carbon | Science of the Total Environment | Antibiotics: Metronidazole, Sulfamethoxazole | chemical activation with k2co3 |  | Experimental design applied | [13] |
| optimization of biosorption potential of nano biomass derived from WS for the removal of malachite green from liquid solutions | Journal of molecular liquids | Malachite green | chemical activation method using concentrated phosphoric acid |  | contains alogorithmic models for optimization (similar to minitab) | [14] |
| Walnut shell as a potential low cost lignocellulosic sorbent for dyes and metal ions | Cellulose | Dyes: Methylene blue, Methyl violet and Murexide, Heavey metals: Fe3+ and Cu2+ | Chemically modified using acetic acid alone and a mixture of acetic acid and hydrogen peroxide |  | Untreated walnut shell most effective for removal of metal ions. Activated most effective for dye removal | [15] |
| Comparison of the adsorption capacity of organic compounds present in producted water with commercially obtained walnut shell and residual biomass | Journal of environmental chemical engineering | real and synthetic produced water (3% gasoline, 10% Oleic acid, 2% lubricant motor oil, and 85% distilled water) | No activation performed |  | GC of real pw available ionic strength study included | [16] |
| Adsorption of methylene blude dye from aq solutions onto walnut shells prodwer | Surfaces and interfaces | Methylene blue | No activation performed |  | Ionic strength study included | [17] |
| Multifunctional walnut shell layer used for oil/water mixtures separation and dyes adsorption | Applied surface science | Methylene blue, rhodamine B and Crystal violet | No activation performed |  | Ionic strength study included | [18] |
| adsorption of naphthalene from aq solution onto fatty acid modified walnut shells | Chemosphere | Polycyclic aromatic hydrocarbons (PAC) | Fatty acid modified (capric acid, lauric acid, palmitic acid and oleic acid) through esterification in n-hexane with sulphuric acid as catalyst |  | talks about partition coefficient | [19] |
| Adsorption of methyl blue from solution using walnut shell and resuse in a secondary adsorption for congo red | Bioresource technology reports | methyl blue and congo red | No activation performed |  |  | [20] |
| characterization and methanol adsorption of walnut shell activated carbon prepared by KOH activation | Journal Wuhan University of Technology, Materials Science Edition | Methanol | Prepared by KOH activation (chemical activation) |  | Article addresses the effect of changing carbonization and activation temperaures on BET surface and micropore volume | [21] |
| Adsorption of congo red and methylene blue dyes on an ashitaba waste and a walnut shell based AC from aq solutions | Chemical engineering journal | Methylene blue and congo red | Chemical activation with znCl2 |  | reported physical interpretations of dyes on surface of adsorbent (physical modeling ) | [22] |
| Synthesis of walnut shell modified with titanium dioxide and zinc oxide nanoparticles for efficient removal of humic acid from aq solutions | Journal of water and Health | Humic acid | Chemical activation with Zinc Oxide and Titanium Oxide |  |  | [23] |
| Tetracycline and sulfamethoxazole adsorption onto nanomagnetic walnut shell rice husk | International journal of environmental analytical chemistry | Pharmaceutical drugs: Tetracycline and sulfamethoxazole | Nanomagnetic walnut shell - rice husk prepared via calcination and co-deposition. Walnut particles were calcined and magnetized by mixing with FeCl3.6H2O and FeCl2.4H2O |  | Includes suggested adsorptin mechasims | [24] |
| Cr(VI) adsorption by a green adsorbent walnut shell: Adsorption studies, regeneration studies, scale-up and economic feasibility | Process safety and environmental protection | Cr(VI) | No activation performed |  | adsorption columns employed + regeneration with NaOH | [25] |
| nano-magnetic walnut shell-rice husk for Cd(II) sorption | Heliyon | Cd(II) | Chemical activation through calcination and magnetization processes |  | Design of experiments used + adsorption occurred through electrostatic attractive forces | [26] |
| Synthesis, characterization of amino-modified walnut shell and adsorption of Pb(II) ions from aq solution | Polymer bulletin | Pb(II) | Chemically modified with triethylene tetraamine |  |  | [27] |
| Adsorption of lead ion from aq solution by modified walnut shell: Kintetics and thermodynamics | Environmental technology | Pb(II) | Walnut shell was reacted with Maleic anhydride |  |  | [28] |
| Evaluation of lead adsorption kinetics and isotherms from aq solution using natural walnut shell | International journal of environmental research | Pb(II) | No activation performed |  |  | [29] |
| Removal of Pb(II) from aq solution by sulfur functionalized walnut shell | Environmental science and pollution research | Pb(II) | Chemically modified by grafting with sulfur containing functional groups using xanthate |  | The adsorption was the result of a combination of complexation and ion exchange | [30] |
| Performance evaluation of agro-based adsorbents for the removal of cadmium from wastewater | Desalination and watertreatment | Cd(II) | chemically treated with sulfuric acid |  |  | [31] |
| Functionalization of powdered walnut shell with orthphosphoric acid for congo red dye removal | Particulate science and technology | Congo red dye | Chemically activated using orthophosphoric acid |  | Regeneration performed using HCl | [32] |
| Walnut shell supported nanoscale Fe for the removal of Cu and Ni ions from water | Journal of applied polymer science | Cu(II) and Ni(II) | Chemically activated using nano zero valent iron | Cu 458.7  Ni 327.9 |  | [33] |
| Adsorption equilibrium and kinetics of Pb(II) from aq solution by modified walnut shell | Wiley library | Pb(II) | Chemical activation with acrylic acid | 210.14 mg/g | Adsorption process verified by EDS | [34] |
| Removal of 2,4,6-trichlorophenol from aq solutions using agricultural waste as low cost adsorbents | Environment protection engineering | 2,4,6-trichlorophenol | No activation | 27.18 | Ionic strength study included | [35] |
| Batch and column adsorption of reactive red 198 from textile indutry effluent by microporous activated carbon developed from walnut shell | waste and biomass valorization | reactive red 198 | Chemical activation using phosphoric acid and potassium hydroxide | 79.15 |  | [36] |
| Treatment of wastewater containing crystal violet using walnut shell | Journal of residuals science and technology | Cyrstal violet | No activation | 714.3 |  | [37] |
| Adsorption oxidation of hydrogen sulfide of Fe/walnut-shell activated carbon surface modified by NH3 plasma | Journal of environmental sciences | H2S (g) | Prepared by coprecipitation where walnut shell was mixed will colloid solution Fe(NO3)3.9H2O and Na2CO3 solution. afterwards, solution was mixed with KOH |  | Non-thermal plasma is an effective method to improve the surface properties of walnut shell. Other methods also used include heat treatment, microwave and surface oxidation. | [38] |
| Eco-friendly walnut shell powder based facile fabrication of biogenic Ag-nanodisks and their interaction with bovine serum albumin | Journal of photochemistry & photobiology | Juglone a yellow brown dye | No activation |  | No adsorption occurred. Extraction. | [39] |
| Biochar produced from the co-pyrolysis of sewage sludge and walnut shell for ammonium and phosphate adsorption from water | Journal of environmental management | NH4+ and PO43- | co-pyrolysis of sewage sludge and walnut shell at 873 C | ammonium: 22.85  Phosphate: 303.49 |  | [40] |
| Removal of Cr(VI) from aq solutions by using activated carbon supported iron catalysts as efficient adsorbents | World journal of engineering | Cr(VI) | Activated carbon was prepared by pyrolysis and physical activation | 29.673 mg/g | The adsorbent was Fe impregnanted. 4 different temperatures were studied (300, 400 and 500 C) for calcinations/ TGA curve of raw walnut shell available. Optimum pyrolysis temperature is 900 C | [41] |
| Removal of brilliant green and crytal violet from mono and bi component aq solutions using NaOH modified walnut shell | Analytical and bioanalytical chemistry research | Dyes: Brilliant green and Crystal violet | NaOH-modified walnut shell | BG: 146.4 mg/g, CV: 123.2 mg/g | multivariate calibration methods such as partial least squares expand the applicability of spectrophotometric methods for multi-component analysis | [3] |
| Removal of nitrate from synthetic aq solution and groundwater in a continuous pilot system using chemical activated carbon derived from walnut shell | water practice and technology | Nitrate | Chemo-physical activation with 5% H3PO3 | 2.31 mg/g | Continuous mode adsorption studies | [42] |
| Raw walnut shell modified by non-thermal plasma in ultrafine water mist for adsorptive removal of Cu(II) from aq solution | Royal society of chemistry | Cu(II) | Walnut shell was treated with non-thermal plasma in ultrafine water mist | 39.4 mg/g | Include in the physical/chemical activation section in the review paper. | [43] |
| Removal of Cu(II), Zn(II) and Cd(II) ions from aq solutions by adsorption on walnut shell equilibrium and thermodynamics studies: treatment of effluents from electroplating industry | Desalination and watertreatment | Cu(II), Zn(II) and Cd(II) | No activation | Cu : 14.43 mg/g, Zn: 7.47 mg/g, Cd: 7.29 mg/g | More effective at higher pH values andat higher temperatuers | [44] |
| Fixed-bed column and batch reactors performance in removal of diazinon pesticide from aq solutions by using walnut shell-modified activated carbon | Enivronmental technology & innovation | diazinon | Chemical activation using phosphoric acid | 34.98 mg/g | No changes with pH | [4] |
| Highly synergistic effects on ammonium removal by the co-system of pseudomonas stutzeri XL-2 and modified walnut shell biochar | Bioresource technology | Ammonium | A two step modification: 1- walnut shell was pyrolyzed at 300 C and impregnated by 1 M NaOH for 4 hours. 2- the dried material was pyrolyzed at 450 C and impregnated with 0.5 M MgCl2 for 4 hours. | 3.29 mg/g |  | [45] |
| Co-carbonization of biomass and oily sludge to prepare sulfamethoxazole super adsorbent materials | Bioresources | Acid red 18 dye | Carbonization: walnut shells heated to a temperature of 500C for 1 hr. The particles soaked in KOH for 24hrs and then dried at 103C via an electricity heat drum. Activation: KOH impregnated particles were heated to final temperature of 800C for 1 hr and dried at 103C | 79.15 | No pH studies. Adding TiO2 improve specific surface area | [46] |
| Mercury ion adsroption on AC@Fe3O4-NH2-COOH from saline solutions | Desalination and watertreatment | dimethyl amino ethyl azide | chemical activation using ZnCl2 | 161.3 mg/g | Design of experiments implemented | [47] |
| synthesis and characterization of arginine-doped polyaniline/walnut shell hybrid composite with superior clean-up ability for Ch(VI) from aq media | Science of the Total Environment | Sulfamethoxazole | chemical activation using K2CO3 by one-step and two-step activation | 361.9 | information regarding functioncal groups | [48] |
| Artificial neural network and multiple linear regression for modeling sorption of pb ions from aq solutions onto modified walnut shell | Korean journal of chemical engineers | Mercury | Physical activation with water vapor at 800C. Magnetic nano particles were prepared by conventional co-precipitation method. The adsorbent is also amino functionalized | 80 mg/g | Influence of salinity was studied and found to be negligible (pH level between 7 - 11). The addition of oxygen functional containing NH2-COOH groups increases the capacity of adsorbent for removal of positively charged contaminants. pH and salinity most effective parameters | [49] |
| Theoretical and experimental study of cephalexin batch adsorption dynamics using walnut shell-based activated carbon | Journal of molecular liquids | Cr(VI) | The hybrid composite was synthesized by interfacial polymerization of freshly distilled aniline on ws | 562.83 | Design of experiments implemented. Adsorption efficiency decreases with pH. Adsorption mechanism predominantly governed by electrostatic interactions. Conducting polymes worth investigation | [50] |
| Removal of mthylene blue dye from aq solutions by adsorption on levulinic acid modified natural shells | separation science and technology | Pb2+ | esterfication performed via isopropylidene malonate | 192.3 | reported that pHzpc for raw walnut shell is 6.1. efficiency increases with pH. Two different models were used to predict the removal percentage of lead ions uder different experimental conditions. The two models are artificial neural network (ANN) nad multiple linear regression (MLR) | [51] |
| Phosphorus removal from aq solution using modified walnut and almond wooden shell and recycling as soil amendment | Desalination and watertreatment | cephalexin antibiotic | Chemical activation with ZnCl2 | 233.1 |  | [52] |
| Adsorption of basic dyes using walnut shell-based biochar produced by hydrothermal carbonization | International journal of phytoremediation | Methylene blue dye | chemically modified with levulinic acid | 294.1 | three natural shells were used. Walnuts exhibited highest adsorption capacity. The shells were rinsed with NaOH to remove impurities for 1 hour. | [53] |
| Facile synthesis of resuable magnetic Fe/Fe3C/C composites from renewable resources for super-fast removal of organic dyes | Environmental monitoring and assessment | Phosphorus | chemically modified with NaOH, epichlorohydrin and trimethelamine to insert quaternary ammonium groups | 22.73 | two types of natural shells were used, walnut shell exhibited higher adsorption capacity than almond shells | [5] |
| Walnut shell powder as a low-cost adsorbent for methylene blue dye: isotherm, kinetics, thermodynamics, desorption and response surface methodology examinations | Chemical research in chinese universities and springer | Methylene blue and malachite green | Hydrothermal carbon was produced from walnut shells and chemically activated using nitric acid | MB: 192.30, MG: 166.66 | this paper compares the efficiency of raw walnut shell, activated and functionalized biosorbents. hydrothermal carbonization is a thermochemical process to convert lignocellulosic biomass into biochar. The mass ratio of H/C in the biochar decreases with increasing aromaticity. The mass ratio of (N+O)/C which is used as a measure of polarity increases as the polarity increases. in an FTIR analysis, if peak intensities decrease this is due to dehydration | [54] |
| Adsorption of hexavalent chromium modified walnut shell from solution | Powder technology | Methylene blue, methyl red, methyl orange and malachite green | Fe/Fe3C nanoparticles incorporated onto walnut shell. Walnut shell activated through pyrolysis | MB: 641, MR: 500, MG: 500, MO: 500 | After incorporating nanoparticles, surface area+total pore volume+micropore area increased. KOH activation at high temperature (1073 K) created additional pore volumes in the range of 0.7 - 5 nm. Strong van der waals interactions involved in the adsorption of MB. thoroughly explain the mechanism of adsorption | [55] |
| Investigation of effectiveness of pyrolysis products on removal of alizarin yellow GG from aq solution: A comparative study with commercial activated carbon | Scientic reports nature science | Methylene blue | No activation | 36.631 | MB absorbs at 1643 cm-1 in the FTIR analysis. Good FTIR discussion. Good analysis discussion in terms of characterization. MB is a basic dye | [56] |
| Application of powdered activated carbon coated with zinc oxide nanoparticles prepared using a green synthesis in removal of reactive blue 19 and reactive black 5 | Royal society of chemistry | naphthalene and phenanthrene | chemical activation by adding KOH via microwave irradiation | N: 93.62, P: 145.62 | Microwave-assisted irradiation is undoubtedly an alternative and novel heating method to convert biomass into AC | [57] |
| characterization of hydrogen peroxide modified hyrochars from walnut shell for enhanced adsorption performance of methylene blue from aq solution | water science & technology | Cr(VI) | Walnut shell was modified using diethylenetriamine through grafting | 50.1 | Adsorption column studies. Amino modification may be an effective method to promote the adsorption ability of walnut shell. Amino group existing in the surface of adsorbent can bind heavy metal positive ions through the complexation | [58] |
| Sorption of thallium on walnut shells and its enhancement by the lignosulfonate-stabilized gold colloid | water science & technology | Alizarin GG yellow dye | Pyrolysis of biomass was carried out at different temperatures in the range of 400 - 700 C | 4.92 | the surface area of walnut shell adsorbent increased considerably when pyrolysis temperature increased from 300 to 700 C. Temperatures above 700 causes the gasification process rather than the pyrolysis process pHpzc for walnut shell is 4.8 | [59] |
| Application of organic waste for the adsorption of Zn(II) and Cd(II) ions | Desalination and watertreatment | reactive blue 19 and reactive black 5 | floating method used to prepare activated carbon coated with zinc oxide | Blue: 94.33, Black: 71.42 |  | [60] |
| Walnut shell activated carbon: optimization of synthesis process, characterization and application for Zn (II) removal in batch and continuous process | Desalination and watertreatment | Methylene blue | Hydrothermal carbonization of walnut shell and then chemically modified using hydrogen peroxide | 173.92 | hydrogen peroxide modification of hydrochars might have increased the oxygen containing functional groups such as carboxyl groups resulting in more than three times increase of adsorption capacity | [61] |
| A new adsorbent modified from walnut shell for the adsorption of Ni(II) from aq solution | Polish journal of environmental studies | Thallium | No activation | 1.329 |  | [62] |
| Enhancing removal of Cu(II) from aq solution by walnut shell-based activated carbon with pyrolusite modification | Environment protection engineering | Zn(II) and Cd(II) | No activation | Zn: 26.60, Cd: 21.10 |  | [63] |
| Multiparametric filtration effect of the dyes mixture removal with low cost materials | Materials research express | Zn(II) | No activation | 89.25 |  | [64] |
| Microwave-assisted for clean and rapid fabrication of highly efficient magnetically separable activated carbon from agriculture shells for low grade industrial corn syrup decoloration | Desalination and watertreatment | Ni(II) | Adsorbent prepared by grafting aspartic acid onto walnut shell | 568.18 |  | [65] |
| Synthesis of a La(OH)3 nanorod/walnut shell biochar composite for reclaiming phosphate from aq solutions | Internation journal of environmental analytical chemistry | Cu(II) | walnut shell modified with pyrolusite and impregnated with phosphoric acid | 34.4 | Pyrolusite was proved to have significant modification effect in preparation of high-quality walnut shell based activated carbons | [66] |
| surface interactions during the removal of emerging contaminants by hydrochar based adsorbents | Arab journal of basic and applied science | Methylene blue, crystal violet and methl orange | No activation | - | Column study. Removal of dye increased with decrease in particle size | [67] |
| Kinetic and equilibrium study of the removal of reactive dye using modified walnut shell | Food and bioproducts processing | corn syrup | walnuts chemically activated by ZnCl2 + FeCl3 + FeCl2 using by microwave assisted impregnation technique | - | Good characterization analysis. Chemical activation is the prominent method for producing activated carbon in large quantity. Chemical activation favoured over physical. Iron chlorides are good candidate materials for manufacturing the magnetic activated carbon. Chemical activation of agricultural waste by zncl2 and Fecl3 was successfully performed to fabricate microporous ac. | [68] |
| Fabrication of magnetic activated carbon by carbothermal functionalization of agriculture waste via microwave-assisted technique for cationic dye adsorption | Colloids and surfaces: physicochemical and engineering aspects | Phosphate | prior to chemical activation, walnuts were heated in a muffle furnace at a temperature of 400 C. then walnuts were loaded with La(OH)3 | 75.08 | after activation, carbon content decreased, pore volume increased and pore size decreased and BET surface area increased substantially | [69] |
| Preparation of controlled porosity activated carbon from walnut shell for phenol adsorption | Molecules | Emerging contaminants: fluoxetine and nicotinic acid | physical activation through hydrothermal carbonization with water and carbon dioxide | F: 44.1, N: 91.9 | mention adsorption mechanisms | [70] |
| Removal of ochratoxin A from liquid media using novel low-cost biosorbents | water science & technology | Reactive brilliant blue | adsorbent synthesized using solid sodium hyroxide and aspartic acid | 224.42 | Adsorbents regenerated using NaOH. Post functionalizing walnut shells, surface area + total pore volume + average diameter increased tremendously. | [71] |
| Adsorptive removal of methylene blue by fruit shell: Isotherm studies | Advanced powder technology | Methylene blue | Walnuts were carbothermally converted to magnetic activated carbon by a micro-wave decoration of iron oxide nanoparticles onto the shell surface. ZnCl2 and FeCl3 chloride were impregnated on walnut shells | 130 | Minitab software for design of experiments was used. The effect of carbonization time was investigated. The power of microwave power was investigated. Powers above 600 W destruction of carbon matrix occurs. It was reported that at time 30 minutes, maximum adsorption capacity was achieved. Effect of carbonization atmosphere has also been investigated. | [72] |
| Comparison study of naphthalene adsorption on activated carbons prepared from different raws | Desalination and water treatment | phenols | Chemical activation with znCl2 | 214 | Full article not available | [73] |

Table S3. Literature review detailing adsorption studies of heavy metal ions during the period 2016 and 2021 reported in Figure 3.

|  |  |  |
| --- | --- | --- |
| Number of research studies | Heavy metal ion | Reference |
| 7 | Pb | [12], [27 – 30], [34] and [51] |
| 5 | Cu | [15], [33], [43 – 44], and [66] |
| 4 | Cr | [25], [41], [50] and [58] |
| 4 | Cd | [26], [31], [44] and [63] |
| 3 | Zn | [44], [63] and [64] |
| 3 | Ni | [12], [33] and [65] |
| 1 | Tl | [62] |
| 1 | Hg | [49] |

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