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Linking Treatment to Performance: A Review of How Modification Governs Metal and Dye Adsorption on Rice Husk

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Abstract

Water pollution by heavy metals and synthetic dyes is one of the more stubborn environmental challenges affecting both industrialised and emerging nations. Conventional treatment approaches – precipitation, ion exchange, membrane filtration and the like – work well enough, but are often too costly or operationally demanding for small and medium firms. Thus agricultural by-products have continued to be the subject of interest as inexpensive, abundant and mostly biodegradable alternatives. Rice husk is the protective outer layer detached from the grain during milling and is generated in huge amounts every year and is usually thrown or burnt. This review summarises and discusses the literature on the use of rice husk, in its raw and modified forms, for the removal of metal ions and dyes from aqueous solutions. We summarise the physicochemical features that make the material attractive, i.e. its lignocellulosic backbone, its abundant silica and its surface functional groups, and review the main preparation strategies, among them alkali and acid treatment, thermal conversion to biochar, activation into carbons, extraction of silica and assembly of composites. Reported adsorption capacities are compiled for a range of metals, notably Pb, Cd, Cu and Cr, and for both cationic and anionic dyes. The influence of solution pH, adsorbent dose, contact time, initial concentration and temperature is considered, together with the isotherm, kinetic and thermodynamic models most often applied. Regeneration, the practical limitations of the material, and some directions for further study are addressed in closing.

Keywords: *Rice Husk; Biosorbent; Heavy Metals; Dye Removal; Adsorption; Agricultural Waste; Water Treatment.*

I. INTRODUCTION

The discharge of untreated or partially treated effluents from mining, metal finishing, tanning, textile and dyeing operations has loaded both surface water and groundwater with two broad classes of persistent pollutant: heavy metal ions and synthetic dyes. Metals such as lead, cadmium, chromium, copper, nickel and zinc are not biodegradable, tend to accumulate in living tissue, and are toxic even at low concentrations [1]. Dyes, for their part, are visible at very low levels, cut down light penetration in receiving waters, and a number of them or their breakdown products are mutagenic or carcinogenic [1]. Both groups therefore have to be removed before discharge.

A familiar set of treatment options exists: chemical precipitation, coagulation, ion exchange, reverse osmosis, electrochemical methods and adsorption onto activated carbon. Much of this activity, however, has significant downsides – high reagent or energy costs, inadequate removal at trace levels, or production of secondary sludges that themselves need disposal [2]. Adsorption is quite simple, adaptable and effective; the challenge is that commercial activated carbon is costly and this has inspired researchers to seek less expensive alternatives from natural materials and waste streams [1].

Agricultural residues are especially appealing in this respect. They are renewable, generated in large volumes, and otherwise of little commercial value. Rice husk is a prime candidate. As the hard sheath enclosing the rice grain, it is separated during milling and accounts for roughly one fifth of the paddy mass; with global paddy output now well above 750 million tonnes a year [3], the quantity of husk available is immense. Much of it is simply burned or dumped, which creates a disposal problem of its own. The present overview brings together the literature on rice husk — raw, treated, carbonised and composited as a sorbent for metals and dyes, and tries to offer a balanced reading of what the material can, and cannot, do.

II. RICE HUSK: AVAILABILITY, COMPOSITION AND SURFACE CHARACTERISTICS

Rice (Oryza sativa) is among the most widely grown crops on the planet, and the bulk of production is concentrated in Asia, with China, India, Indonesia, Bangladesh, Vietnam and Thailand together dominating the output [3]. Because husk represents about 20% of the harvested paddy by weight, tens of millions of tonnes are generated each year as a milling by-product [3]. It has been seldom used in the past due to low bulk density, abrasiveness and high ash content, so that a major share has rotted or been burnt in the open with emission of particles and greenhouse gases.

It is a chemical lignocellulosic material. Compositions vary with cultivar, climate and soil but most analyses place cellulose in the region of 25-35%, hemicellulose at roughly 18-25% and lignin near 15-25% with the remaining made up largely of silica mixed with tiny amounts of protein and soluble matter [1]. The husk is different from many other biomass in that there is a very high amount of silica. The rice husk ash from burning can be 85-95% amorphous silica [4]. Cellulose, hemicellulose and lignin combined give a number of oxygen containing functional groups-hydroxyl and carboxyl and after treatment carbonyl and silanol-which operate as binding sites for metal cations and charged dye molecules [1].

The unmodified husk surface has a moderate specific surface area, and it is relatively unreactive with poor raw adsorption capability. Nevertheless, the honeycomb-like and porous structure, together with the silica skeleton and exchangeable surface groups, provides a useful initial framework which can be opened up and chemically modified by adequate treatments [4]. The substance is nevertheless of interest to scientists since it is cheap, abundant and easy to modify.

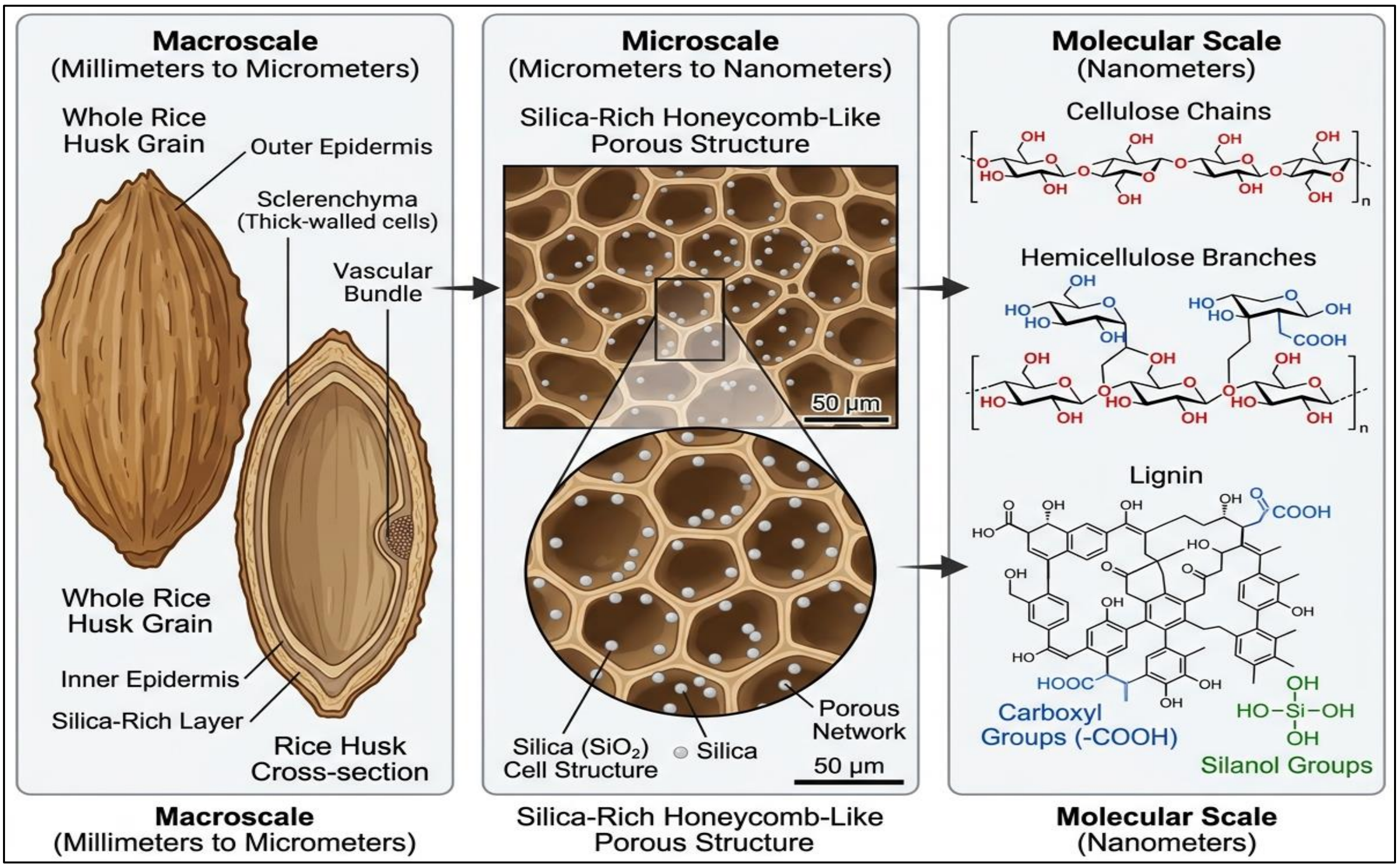

Fig 1 Schematic Illustration of the Hierarchical Structure and Ridged, Silica-Rich Surface of rice Husk, Showing the Surface Groups Available for Binding.

Table 1 Typical Chemical Composition of Rice Husk Reported in the Literature (wt.%).

Component	Content (wt.%)
Cellulose	25–35
Hemicellulose	18–25
Lignin	15–25
Silica (SiO_2)	15–20
Ash	13–23
Moisture and soluble matter	2–8

Typical ranges compiled from the literature; on combustion the resulting ash is 85–95% amorphous silica [1,4].

III. PREPARATION AND MODIFICATION OF RICE HUSK ADSORBENTS

Various different techniques have been investigated for the conversion of rice husk to a functional sorbent and vary substantially in cost, complexity and the nature of the substance produced.

The simplest approach is to work with raw or washed husk: the husk is rinsed with water (sometimes hot water or dilute acid) to strip away dust and soluble organics, dried, and ground to a chosen particle size. This costs almost nothing and avoids chemical inputs, but the capacities obtained are generally low, and untreated husk can leach colour and organic matter into the treated water, raising its chemical and biological oxygen demand [2]. For this reason most workers treat raw husk as a baseline against which the modified forms are judged.

Chemical modification is the most common upgrading step. Alkali treatment — typically with sodium hydroxide — removes part of the lignin and hemicellulose, exposes cellulose hydroxyl groups and roughens the surface, which usually raises the uptake of both metals and cationic dyes; acid treatment with HCl, sulfuric or citric acid can introduce or expose carboxyl functionality and clean out the pore structure. Working with lead, Dada and co-workers prepared modified rice husk and analysed Pb(II) uptake through a set of isotherms, reporting favourable, Langmuir-type behaviour [5]. For cadmium, treating the husk with sodium hydroxide raised the Langmuir capacity from about 5.1 mg/g for the untreated material to roughly 11 mg/g for the treated form, with the best performance near neutral pH [6]. Grafting and functionalisation push this idea further: phosphorylation, amine grafting and the attachment of quaternary ammonium groups have all been used to steer selectivity towards particular ions or dye classes.

Thermal conversion gives a different product. Pyrolysis of husk under limited oxygen yields rice husk biochar, a carbon-rich, porous solid whose yield, surface area and surface chemistry depend strongly on the pyrolysis temperature, the heating rate and the residence time [4]. Native husk biochar often has only a modest surface area, but it can be improved substantially; hydrothermal alkali activation, for example, lifted the surface area of one husk biochar from about 22 to over 430 m^2/g and produced a mesoporous material with a malachite green capacity of around 373 mg/g [7].

A further step is activation into carbon. Carbonisation followed by chemical activation, commonly with KOH, $ZnCl_2$ or H_3PO_4, or by physical activation with steam or CO_2, develops a much larger and more uniform pore network. Rice husk activated carbons prepared by KOH activation have been applied to methylene blue with high efficiency, the equilibrium data conforming to the Langmuir model and the kinetics to a pseudo-second-order rate law [8,9]. Because the ash is so rich in silica, controlled combustion and alkaline extraction can also recover relatively pure silica from the husk [4], which in turn serves as a low-cost precursor for ordered mesoporous silicas used in dye capture.

Finally, husk and husk-derived carbons have been combined with metal oxides, layered double hydroxides and magnetic phases, either to add functionality or to allow easy magnetic separation. A magnesium oxide–rice husk biochar composite, for instance, showed enhanced Cd(II) uptake relative to the plain biochar [10], and a CuAl layered double hydroxide / husk biochar composite improved the removal of a cationic dye [11].

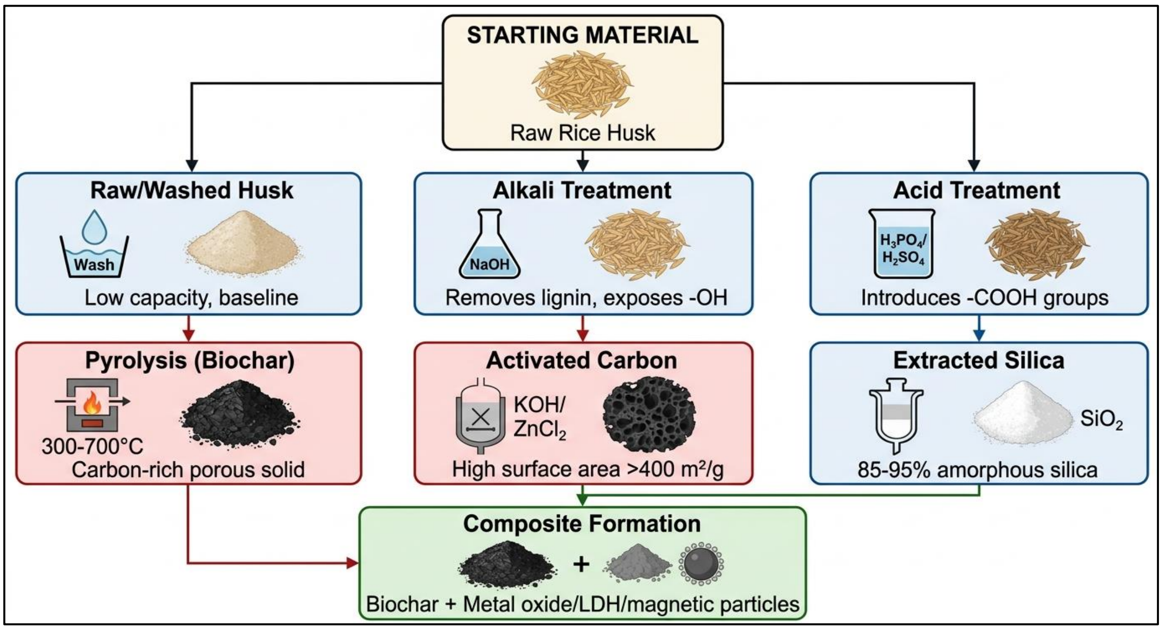

Fig 2 Principal Routes for Preparing Rice Husk–Based Adsorbents: Raw/Washed Husk, Alkali- or Acid-Treated Husk, Biochar, Activated Carbon, Extracted Silica, and Composites.

Table 2 Common Modification Methods for Rice Husk and Their Reported Effect on Adsorption Performance.

Method	Reagent / conditions	Reported effect	Ref.
Raw / washed husk	Water or dilute-acid wash, drying, milling	Baseline; low capacity; possible colour / COD leaching	[2]
Alkali treatment	NaOH	Removes lignin & hemicellulose, exposes –OH; Cd uptake ~5.1→11 mg/g	[6]
Acid treatment	H_3PO_4, H_2SO_4, citric acid	Introduces / exposes –COOH; Pb up to ~139 mg/g	[5]
Pyrolysis (biochar)	300–700 °C, limited O_2	Carbon-rich porous solid; properties set by temperature	[7]
Hydrothermal alkali activation	NaOH, hydrothermal	Mesoporous; SSA ~22→435 m²/g; MG 373 mg/g	[7]
Chemical activation (carbon)	KOH, $ZnCl_2$, H_3PO_4	High surface area; efficient MB removal	[8,9]
Composite formation	Metal oxide, LDH, magnetic phase	Extra binding sites; magnetic separation	[10,11]

SSA = specific surface area; MG = malachite green; MB = methylene blue.

IV. REMOVAL OF HEAVY METALS

Most of the priority metal contaminants have been examined in the literature for rice husk and its derivatives, lead and cadmium being the most studied.

Lead is regularly one of the most tightly bonded ions, a characteristic commonly ascribed to its relatively high ionic radius and its strong affinity for surface groups containing oxygen. The modified husk exhibited Langmuir behaviour for adsorption of Pb(II) [5] and the husk-derived biochars lifted the values much higher; the wider literature on modified biochars shows that surface engineering is the major lever for enhancing both lead and cadmium capacities [12].

Due of its toxicity and mobility in the environment, cadmium has been investigated extensively. Untreated husk generally gives capacities of only a few mg/g but alkali treatment doubles this - from about 5 to 11 mg/g in one careful equilibrium and kinetic study, which also found the process to be endothermic and spontaneous and well described by the Langmuir isotherm and a pseudo-second-order rate law [6]. Loading the biochar with reactive phases raises the performance further; the magnesium oxide composite mentioned earlier is a clear example [10].

Beyond lead and cadmium, husk-based sorbents have been applied to copper, chromium, nickel and zinc, though usually with somewhat lower or more variable capacities. Copper(II), like the other divalent cations, is taken up mainly through interaction with oxygen-bearing groups and shows the familiar pH dependence. Chromium presents a more complicated picture, because in solution it exists both as Cr(III) and as the more toxic, oxyanionic Cr(VI); removal of Cr(VI) on raw husk is typically modest and is favoured at low pH, where the protonated surface can attract the negatively charged chromate and dichromate species, and it often involves partial reduction to Cr(III) [2]. Nickel and zinc are generally bound less strongly than lead, a trend that broadly tracks differences in ionic properties and hydration energy [2]. The general lesson from the agricultural-waste literature is that, while raw husk can remove all of these metals to some degree, useful capacities for the more weakly bound ions usually require chemical or thermal upgrading [1,2].

The binding of metals to rice husk is rarely governed by a single mechanism. Ion exchange — the release of H^+, Ca^{2+} or Na^+ as the metal is taken up complexation with carboxyl, hydroxyl and silanol groups, electrostatic attraction to a negatively charged surface, and, at higher pH, a degree of surface precipitation all contribute, their relative weight depending on the metal, the modification and the solution conditions [2]. FTIR and SEM–EDS analyses before and after adsorption are routinely used to evidence these interactions, typically showing shifts in the O–H and C=O bands and the appearance of the metal on the loaded surface [6].

Table 3 Reported Maximum Adsorption Capacities of Rice Husk–Based Adsorbents for Selected Heavy Metals.

Adsorbent	Metal	Capacity (mg/g)	Conditions	Ref.
Raw rice husk	Cd(II)	5.13	pH 7	[6]
NaOH-treated rice husk	Cd(II)	11.06	pH 7	[6]
H_3PO_4-modified rice husk	Pb(II)	138.89	C_0 100 mg/L	[5]
H_3PO_4-modified rice husk	Zn(II)	101.01	—	[13]
H_3PO_4-treated rice husk	Cu(II)	17.04	pH 4	[14]
Rice husk (functionalised)	Cr(VI)	379.63	acidic pH	[15]

Values are Langmuir monolayer capacities (q_{max}) reported under the cited conditions.

V. REMOVAL OF DYES

Dyes are traditionally classified as cationic (basic) and anionic (acid, reactive, direct), and rice husk behaves considerably differently towards the two a distinction that the early overview by Chuah and colleagues already highlighted [1].

The most studied group are cationic dyes, especially methylene blue and malachite green. Activated carbons made from husk are efficient removers of methylene blue with equilibrium data fitting the Langmuir model and kinetics following a pseudo-second order equation [8,9]. In the case of chemically modified husk, malachite green was studied, and a complete thermodynamic analysis stated the uptake as endothermic and spontaneous [16]. Engineered mesoporous husk biochar was also studied, achieving a capacity of around 373 mg/g, and removing over 97% of the dye [7]. Composite materials broaden the field even more, such as the CuAl layered double hydroxide / husk biochar composite [11] prepared for the elimination of a cationic dye. Other basic dyes such as crystal violet and methyl violet have likewise been treated with alkali-modified husk to good effect.

Anionic dyes are intrinsically harder for rice husk to capture, since at most working pH values the husk surface carries a net negative charge that repels the dye anion. The common remedy is to reverse the surface charge by grafting cationic functionality quaternary ammonium groups, for instance which can turn an indifferent sorbent into an effective one and yields high capacities for dyes such as Congo red [1]. A second, rather elegant, technique takes advantage of the silica present in the husk: silica extracted from rice husk ash [4] can be transformed into ordered mesoporous materials, with large accessible surface area and a surface chemistry that can be tailored, making them suitable for the removal or the photocatalytic degradation of a number of dyes. These silica-based alternatives are more complicated to prepare than basic treated husk, but demonstrate how fully the same raw material can be re-purposed.

The distinction between the two dye classes is very significantly pH dependent. The pH can be increased to deprotonate the surface and encourage uptake of cationic dyes, or decreased to protonate the surface and favour anionic dyes [1]. Generally the relevant interactions are electrostatic attraction, hydrogen bonding, π–π interactions with aromatic rings and simple pore filling in the more porous carbons, and one or another of these interactions dominates depending on the dye and the adsorbent [16,7].

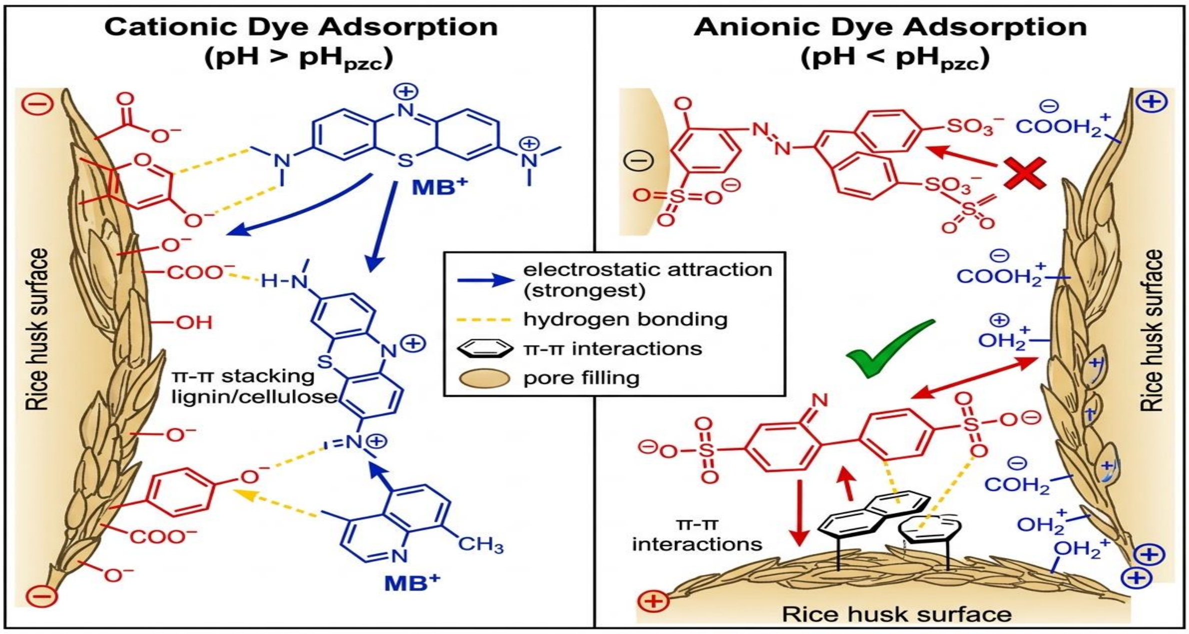

Fig 3 Schematic of the Principal Interactions Governing Cationic Versus Anionic Dye Uptake on Rice Husk Surfaces.

Table 4 Reported Maximum Adsorption Capacities of Rice Husk–Based Adsorbents for Selected Dyes.

Adsorbent	Dye (class)	Capacity (mg/g)	Ref.
H_2SO_4-activated rice husk carbon	Crystal violet (cationic)	64.88	[17]
Xanthated rice husk	Crystal violet (cationic)	90.02	[18]
Multi-step modified rice husk	Methyl violet (cationic)	154.49	[19]
Multi-step modified rice husk	Malachite green (cationic)	996.97	[19]
Mesoporous rice husk biochar	Malachite green (cationic)	373.02	[7]

Bare rice husk has low affinity for anionic dyes; cationic functionalisation (e.g. quaternary-ammonium grafting) is needed for appreciable anionic-dye uptake (see Section 5 and Figure 3).

VI. OPERATING VARIABLES GOVERNING ADSORPTION

The performance of rice husk is controlled by a fixed set of operational variables, which are important for the interpretation of laboratory results and for any future scale-up.

The solution pH is almost without exception the most important element, since it determines both the protonation state of the husk surface and the speciation or charge of the adsorbate. For metal cations and for cationic dyes, increasing pH deprotonates surface hydroxyl and carboxyl groups, making the surface more negative, and so increasing electrostatic attraction and uptake. The practical ceiling is set by the point at which metals begin to precipitate as hydroxides, beyond which apparent removal no longer reflects true adsorption [6,8]. With anionic dyes the reverse is the case, the best uptake is obtained at acidic pH values, when the surface is positively charged [1].

The adsorbent dosage usually exhibits a two-sided behaviour. As mass of husk increases the percentage of pollutant eliminated increases simply because of the availability of more binding sites but the amount adsorbed per gram decreases normally, because at high dose many sites are not used and they may partially overlap or agglomerate [6].

The equilibration speed depends on the contact time. Initially uptake is often brisk when the surface is substantially bare and then slows to a plateau as the accessible areas fill. Equilibrium times reported range from a few minutes for some activated carbons to several hours for raw husk [8].

The initial adsorbate concentration influences the capacity and efficiency both. Increasing the concentration results in a higher driving force for mass transfer and hence a higher loading per gram, but the proportion removed tends to fall as the accessible sites become saturated [6]. Temperature is a function of the thermodynamics of the particular system. Many husk-metal and husk-dye interactions are endothermic so that capacity increases somewhat with temperature [16,6]. A few of them are exothermic and behave diametrically opposite. Other variables – such as the particle size of the husk, which impacts the availability of surface, and the ionic strength of the solution, where competing ions may inhibit uptake – are also important, although less consistently observed.

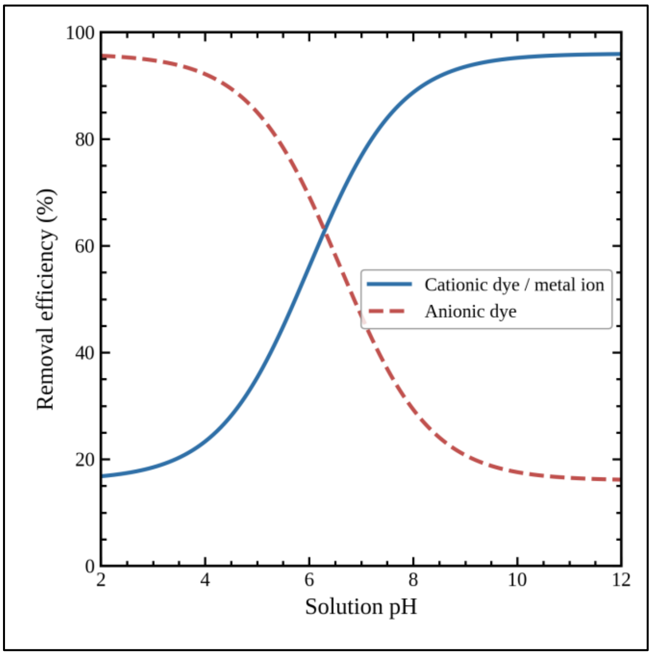

Fig 4 Representative Effect of Solution pH on the Removal of a Cationic and an Anionic Adsorbate by Rice Husk.

VII. EQUILIBRIUM, KINETIC AND THERMODYNAMIC MODELLING

Equilibrium adsorption data are almost always interpreted through isotherm models. The Langmuir model [20], which assumes monolayer coverage on a surface of energetically equivalent sites, is the one most frequently found to give the best fit to rice husk data, and its maximum-capacity parameter offers a convenient basis for comparison between materials [8]. The Freundlich model [21], an empirical expression suited to heterogeneous surfaces and multilayer adsorption, is commonly tested alongside it, while the Temkin [22] and Dubinin–Radushkevich [23] models appear less often; the mean adsorption energy obtained from the latter is sometimes used to argue whether the process is physical or chemical in nature.

Kinetic data are most often described by the pseudo-first-order equation of Lagergren [24] and the pseudo-second-order equation of Ho and McKay [25]. In the large majority of rice husk studies the pseudo-second-order model fits best, which is generally taken to imply that chemisorption — the sharing or exchange of electrons between adsorbate and surface — controls the rate [8,6]. To test whether diffusion inside the particle limits the overall rate, the intraparticle diffusion model of Weber and Morris [26] is frequently applied; a plot that does not pass through the origin is read as evidence that more than one step contributes to the kinetics.

Thermodynamic analysis, finally, yields the changes in Gibbs free energy, enthalpy and entropy that accompany adsorption. A negative free-energy change indicates a spontaneous process, the sign of the enthalpy change distinguishes endothermic from exothermic behaviour, and the entropy term reflects changes in disorder at the solid–liquid interface [16,6]. A word of caution is in order: many of these conclusions rest on linearised forms of the models and on the coefficient of determination alone, and the same dataset can appear to favour different models depending on how it is transformed, so non-linear fitting together with proper error analysis is increasingly preferred.

Table 5 Isotherm, Kinetic and Thermodynamic Models Commonly Applied to Rice Husk Adsorption Data.

Model	Category	Remark	Ref.
Langmuir	Isotherm	Monolayer on identical sites; gives the maximum capacity	[20]
Freundlich	Isotherm	Heterogeneous surface / multilayer (empirical)	[21]
Temkin	Isotherm	Accounts for adsorbent–adsorbate interactions	[22]
Dubinin–Radushkevich	Isotherm	Mean sorption energy → physi- vs chemisorption	[23]
Pseudo-first-order	Kinetic	Lagergren rate equation	[24]
Pseudo-second-order	Kinetic	Chemisorption-controlled; usually the best fit	[25]
Intraparticle diffusion	Kinetic	Weber–Morris; tests diffusion limitation	[26]
$\Delta G°$, $\Delta H°$, $\Delta S°$	Thermodynamic	Spontaneity, endo-/exothermicity, entropy change	[16,6]

Across most rice husk systems the equilibrium follows the Langmuir model and the kinetics follow the pseudo-second-order model.

VIII. REGENERATION, REUSE AND PRACTICAL CONSIDERATIONS

For a low-cost sorbent to be genuinely economical, it should ideally be regenerable and reusable over several cycles. Metals are most often stripped from loaded husk with dilute mineral acids such as hydrochloric or nitric acid, which protonate the binding sites and release the cation; dyes may be desorbed with alkali, with organic solvents, or in some cases by oxidative treatment, depending on how strongly and by what mechanism they are held [1,2]. Capacity tends to fall gradually over successive cycles as sites are lost or the structure degrades, so there is a practical limit to how often the material can be reused.

Most of the described work has been done with synthetic single component solutions in batch mode operationally. Some investigations on fixed-bed columns more representative of continuous treatment and real industrial effluents with competing ions and dissolved organics are available, although they are relatively few [12]. Also the fate of the spent sorbent is to be considered: regulated burning can, on the one hand, remove the adsorbed organics, and recover a silica-rich ash, completing part of the loop, but the disposal of metal-laden residue needs to be managed with care.

IX. CHALLENGES AND FUTURE PERSPECTIVES

There are a number of holes in the present work. Protocols are not standardised Particle size, dose, contact time and the manner capabilities are reported change from research to study, which makes direct comparison between publications problematic and often misleading. As stated above, the strong dependence on batch tests with synthetic solutions restricts the extrapolation of the results to the plant scale treatment of real wastewater . The selectivity in multi-component systems, where several metals or a mixture of dyes and salts compete for the same sites, is still not well known and would benefit from rigorous investigation. Mechanistically, using state-of-the-art spectroscopy in combination with computational approaches such as density functional theory offers the possibility to advance the field from the largely phenomenological isotherm-and-kinetics description which presently dominates, to a more detailed picture of how individual functional groups bind specific adsorbates. Finally, life-cycle and cost evaluations that are honest about the chemicals, energy and water spent during modification are required before any claim of low cost or sustainability can be taken at face value [1,12,2].

X. CONCLUSION

Based on the research discussed here, rice husk looks like a credible low-cost biosorbent for heavy metals and dyes. The reasons for its attraction are a lucky combination of features. It is produced in huge quantities as a milling remnant, it is cheap and renewable, and its lignocellulosic-cum-silica structure has binding sites that can be enriched by relatively simple treatments. Raw husk itself is a rather poor sorbent, but alkali or acid treatment, conversion into biochar or activated carbon, extraction of silica and composite production all significantly enhance performance - in the best cases to levels that rival conventional adsorbents. The adsorption of the systems obeyed the Langmuir isotherms and pseudo-second order kinetics well and the solution pH was the most relevant operating variable. What is still mostly missing is the translational work: standardised testing, column and pilot studies, evaluation on real effluents and a clear-eyed cost assessment. Until then, rice husk should be considered a prospective and extensively studied possibility, not a foregone conclusion.

➢ *Highlights*

- Rice husk is an abundant, low-cost agricultural residue well suited as a biosorbent for water treatment.
- Raw husk shows only modest capacity, whereas chemical and thermal modification markedly improves uptake.
- The material removes a wide range of heavy metals as well as both cationic and anionic dyes.
- Adsorption commonly follows pseudo-second-order kinetics and a Langmuir-type isotherm.
- Solution pH is the single most influential operating variable across most husk–adsorbate systems.

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