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Review

Recycling Phosphorus from Agricultural Streams: Grey and Green Solutions

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Abstract: Many intensively farmed soils show high phosphorus (P) contents compared to the thresholds required for agricultural production; 0.084 Mt of P year^{-1} is leaving the European terrestrial system. This paper focuses mainly on non-point flows of P and provides an overview of the most promising and sustainable solutions for P recycling, centred on waste materials from agriculture. Given the global shortage of the primary resource of P, its management is critical for its efficient use. Nowadays, wastage and loss at every stage of the P cycle raise concerns about future supplies and especially about the resulting environmental problems, such as the eutrophication of surface water bodies and the reduction of biodiversity. Recovering P costs more than EUR 640 per tonne depending on the type of technique used. The opportunity for P recovery with green and sustainable technology is, therefore, a great challenge for the next years. Waste materials or by-products of agricultural processing have been considered ecologically safe, low-cost, and highly selective with high pollutant adsorption capacities, which would enable sustainable P recovery, both environmentally and economically. A realistic threshold for considering the reuse of P sustainably at the farm level is EUR 320 per tonne.

Keywords: soil; fertiliser; by-product; surface water; agricultural waste; nutrient recovery

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1. Introduction

The continuing increase in the world's population poses a crucial risk to environmental emergencies, such as the supply of finite (limited) raw materials such as phosphorite, from which phosphorus (P), a key nutrient for ensuring global food support, is derived [1].

In addition, the increasing demand for commodities has intensely augmented attentiveness to their recycling and reuse; no current production process scheme is imaginable without considering the by-products, residues, and wastes [2]. Phosphorus (P) is a crucial element for producing crops and is widely used in both recycled manure and inorganic fertiliser [3,4]. Its cycle has a high impact on the total environment, interfacing the hydrosphere and the pedosphere, and being heavily dependent on the biosphere and anthroposphere. Today, the global society faces serious challenges given the scarcity, importance, unequal global distribution, and, at the same time, regional excess of P. Phosphorite, the primary resource from which P is extracted, is concentrated in a few areas of the globe, with China, Morocco, the United States, Jordan, and Saudi Arabia being the leading producers, followed by ongoing capacity expansion projects in Brazil, Kazakhstan, Mexico, Russia, and South Africa [5,6]. The approximate annual global consumption for P is 3 kg per capita, which has been rising over time for most regions [7]. However, the amounts of P available and price volatilities, as occurred in 2008, have given rise to concerns about future supplies and water and soil pollution, encouraging moves toward the recycling of P [8].

Ninety per cent of extracted P is used for food production [9], mostly indirectly as fertiliser. Therefore, food production depends on a non-renewable resource, which, in recent decades, has become progressively expensive due to the growing global demand, with negative environmental impacts caused by constant mining [10–12].

Europe is the most import-dependent region, with 86% of P's total demand [13]. In EU agriculture, 1.1 million t of P fertiliser was used, a slight reduction of 6.5% since 2012 [14]. Waste deriving from industrial processes, such as sludge, contains agronomical valuable P, so its application on cropland might decrease the necessity of mined fertilisers, but its riskiest contraindication is the content of potentially harmful organic and inorganic contaminants [15]. Given this dependence on P and the global shortage of the primary resource of P, management is critical for the efficient use of P resources. Considering that P management might not be low-cost but is the only option to maintain P supply, as P is not replaceable or renewable, we should be investing in sustainable strategies that will, on the one hand, increase productivity in the short term and, on the other hand, could be a probable low-cost guarantee for the future, supporting the food supply and farmers and safeguarding water quality [16]. The Raw Materials Initiative of the European Commission addresses challenges related to access to raw materials by identifying materials with a high supply risk and economic importance, to which reliable and unhindered access is critical for industry and value chains (Table 1); they set the fundamental importance of phosphate rock. The list should help boost European production of critical raw materials by strengthening recycling activities and, if necessary, facilitating the launch of new recycled materials in line with environmental sustainability and the circular economy. Additionally, it provides a better understanding of how the security of the supply of raw materials can be ensured through diversification, the use of different geographic sources, mining, recycling, or substitution. Critical raw materials are a priority in the Circular Economy Action Plan of the European Union, which aims to promote their efficient use and recycling [17].

Table 1. Major world producers and supply sources of phosphorus and phosphorite, both considered essential raw materials. Data from European Commission [17]. Percentages refer to major producers/importers only.

Raw Materials	Main World Producers (Average 2010–2014)	Main EU Importers (Average 2010–2014)	EU Source of Supply (Average 2010–2014)	Import Reliance Rate *
Phosphorite	China (44%) Morocco (13%) United States (13%)	Morocco (31%) Russia (18%) Syria (12%) Algeria (12%)	Morocco (28%) Russia (16%) Finland (12%) Syria (11%) Algeria (10%)	88%
Phosphorus	China (58%) Vietnam (19%) Kazakhstan (13%) United States (11%)	Kazakhstan (77%) China (14%) Vietnam (8%)	Kazakhstan (77%) China (14%) Vietnam (8%)	100%

(*) The "Import Reliance Rate" takes into account global supply and actual EU sourcing in the calculation of Supply Risk, and it is calculated as follows: EU net imports/(EU net imports + EU domestic production).

Phosphate rock is a finite resource for all practical purposes, subject to price volatility, with varying quality and unequal access across the globe. Additionally, although there is an overall vagueness as to when the peak will be reached, there is a large consensus that the remaining phosphate rock reserves are declining [18]. According to USGS data (2022), almost 220 million t of phosphate rock was extracted in 2021 from global mineral reserves, but only about 0.03 million t were marketable. Of this marketable phosphate rock, consumers used more than 99% in the same year for an estimated EUR 1940 million in sold products. Marketable phosphate rock contains less than 18% P. Therefore, the geopolitical situation can have a significant influence on the availability and price of P; the other dimensions of this issue (price, quality, access) seem more compelling to us currently. Thus, the growing perception of a global P crisis could lead to serious international tensions due to both the distribution of terrestrial reserves and their control, as well as global population growth [19]. According to the European Sustainable Phosphorus Platform (ESPP), the annual world market for phosphate fertilisers is around EUR 45–60 billion. At the time of writing, the price of rock phosphate is EUR 320 per metric ton (+85% February to September

2022) (from https://www.indexmundi.com/commodities/ accessed on 20 November 2022), while diammonium phosphate exceeds EUR 1000 per ton, reflecting the fertiliser end-user price (from https://www.agrarmarkt-nrw.de/duengermarkt.shtm, accessed on 20 November 2022). These are strong arguments for pursuing a circular P economy now, regardless of the exact timetable of phosphate rock resource depletion. Given these issues, P recovery strategies represent the solution to collapsing fertiliser supplies and the growing problem of eutrophication. Economically, P recovery is also beneficial when compared to the potential economic loss due to its release [20].

The current literature on P reuse and recycling has increased dramatically in the recent decade, according to both Scopus and Web of Knowledge, and Elsevier's and Clarivate's abstract and citation databases. This review describes the current industrial alternatives for recovering lost P (grey removal) and proposes bio-based solutions for P recycling (green removal). P recycling from both non-point sources and point sources, such as urban and livestock wastewater, focusing on the reuse of materials derived from agriculture waste, would result in lower costs and environmental impact and could lower P excess.

2. P in the Soil Environment

2.1. Overfertilised Soils

The presence of P is essential for modern agriculture. However, fertiliser efficiency varies between regions, and in general, less than 20% of the P absorbed by the plants is then harvested [21]. Globally, farmers apply about 25 Mt P year^{-1}, and about 14 Mt P year^{-1} is not used by crops, becoming a pollutant. This means that more than half is lost to the environment and can create ecological imbalances in ecosystems and water bodies. Therefore, it is crucial to provide crops with the correct amounts of fertilisers to avoid excesses [22]. In Europe, Panagos et al. [23] estimated the total P in agricultural topsoil (0–20 cm) at a mean of 1412 kg ha^{-1}. A high soil P concentration, conferring no direct agronomic advantage, has resulted in inefficient resource use [24]. When considering European agricultural soils (171 million ha; not considering set-aside agricultural land), P input with inorganic fertilisers is estimated to be a total of 1.3 Mt P year^{-1}. Of which about 11% is estimated as a P year^{-1} surplus, and total P losses in river basins and outlets have been estimated to be about 0.1 Mt P year^{-1} [23]. Therefore, best practices should be to estimate the recommended threshold values for soil P testing (STP), a good indicator for the potential risk of phosphorus movement at the edge-of-field into downstream waters, based on routine soil sampling and analysis that have been identified to help assess the likely yield response to P applied on-farm [25]. However, many regions with intensive agriculture have STP values above the threshold values required for optimal agricultural production [26–28].

2.2. P Losses

As a fundamental element of plant nutrition, P excess does not cause problems for the crop itself but exposes the environment to the risk of P leakage and the consequent eutrophication of water bodies [1,29]. Although we can consider transfer into the oceans a natural process resulting from erosion and runoff, it is nevertheless accelerated by human activities such as arable farming, concentrated animal husbandry, and direct anthropogenic discharges, with losses in the range of 19–31 Mt P year^{-1} [30]. Total P losses to European river basins and sea outlets are estimated to be around 100,000 t P year^{-1} [23]. In general, preventing nutrient losses is more beneficial in energy and economic terms than recycling [31].

Losses from agricultural soils occur in both dissolved and particulate forms, and their transport depends on the soil type, the extent of soil P accumulation, erosion vulnerability, and hydrological connectivity to the waterbody [32–36]. For instance, a negligible decrease in pH is enough for the mobilisation of significant quantities of P [37]. Several studies have shown the influence of catchment characteristics and changes in land use on water quality and the integrity of ecosystems downstream (Table 2).

Agricultural areas play an important role in P losses, as they are the main areas subject to erosion, which facilitates the loss of significant P flows. Their impacts are obvious not only on a local scale but also on a much larger scale [38–41]. Thus, the goal of eutrophication control would be more achievable if P concentrations in soils were kept at or below the recommended threshold values for improved fertiliser response [4,40,42–46], including strategies to mitigate the transfer of P by erosion [47].

Mockler et al. [48] calculated 0.39 kg ha^{-1} as the annual average value of P export to the Irish national territory, of which 51% comes from wastewater and 49% from grazing and agricultural land (0.19 kg ha^{-1}). Van Dijk et al. [49] suggested that emissions from runoff and erosion to the hydrosphere from the 27 EU member countries account for 41% of total P losses. With over 191 million hectares of agricultural land in Europe, 0.084 Mt of P from runoff and erosion is leaving the system each year, values comparable with those estimated by a recent study by Palagon et al. [23].

Table 2. General characteristics of the catchment area sorted by land use. The quantities of phosphorus lost from soils entering surface waters or the P content in the surface waters of the basin are also reported.

Catchments ID	Coordinates	Soils [a]	Land Use [b]	MAP mm year^{-1}	Clay %	OC [b] g kg^{-1}	pH [a]	P Loss kg ha^{-1}	P Loss mg L^{-1}	Ref [c]
			cereals							
Robe (IR)	53°41′32″ N 9°03′45″ W	PZ, UM	CPAH	1150	19	50	6.0	1.09		[50]
Wye (UK)	52°03′16″ N 3°10′32″ W	PZ, CM	CMGFHP	1000	13	132	5.5	0.52		[4]
Chesapeake (US)	37°31′15″ N 76°06′18″ W	AL, AC, LV	CSWHDP	890	13	25	5.0	0.84		[4]
Paimionjoki (FI)	60°28′02″ N 22°40′56″ E	GL, PZ	CLDA	680	35	19	4.9	0.88		[4]
Odense (DK)	55°13′00″ N 10°18′36″ E	CM, LV	CPAH	580	15	17	6.5	0.75		[50]
Palma del Río (ES)	37°41′ N 5°18′ W	VR	CDWLS	570	47	15	7.2	1.07		[42]
Palma del Río (ES)	37°41′ N 5°18′ W	LV	CDWS	570	22	15	7.8	0.67		[42]
			pasture							
Neagh-Bann (IR)	53°31′04″ N 6°43′34″ W	CM, LV	PPFAL	800	20	30	6.7	0.16		[48]
North-West (IR)	54°36′18″ N 8°01′15″ W	PZ, CM	PPF	1100	20	130	5.4	0.21		[48]
Shannon (IR)	53°16′30″ N 7°57′29″ W	PZ, CM	PPF	750	20	50	6.2	0.16		[48]
West (IR)	53°46′40″ N 9°04′34″ W	PZ, GL	PPF	1100	22	55	6.1	0.21		[48]
South-West (IR)	51°56′51″ N 08°52′11″ W	PZ, GL	PPF	850	18	70	5.3	0.20		[48]
South-East (IR)	52°45′21″ N 6°54′28″ W	PZ, GL	PPF	800	21	40	6.1	0.18		[48]
			forestry							
F26 (SE)	57°13′45″ N 13°38′24″ E	PZ, CM	FAL	1070	5	60	4.3		0.12	[41]
N33 (SE)	56°36′01″ N 13°03′28″ E	CM, LV	FAL	820	21	20	6.2		0.16	[41]
N34 (SE)	56°34′46″ N 13°03′45″ E	CM, LV	FAL	820	14	25	6.8		0.10	[41]
M36 (SE)	56°08′36″ N 13°04′51″ E	CM, FL	FAL	720	26	30	6.3		0.20	[41]
M42 (SE)	55°20′27″ N 13°48′04″ E	FL, PZ	FAL	710	16	50	6.4		0.15	[41]
O18 (SE)	58°26′42″ N 12°54′25″ E	PZ, CM	FAL	660	35	40	4.5		0.50	[41]
C6 (SE)	59°42′48″ N 17°18′52″ E	PZ CM,	FAL	620	42	65	4.8		0.21	[41]
I28 (SE)	57°30′14″ N 18°42′58″ E	FL, CM	FAL	590	20	28	6.5		0.18	[41]
U8 (SE)	59°20′45″ N 16°33′33″ E	PZ, CM	FAL	540	50	45	4.7		0.26	[41]
E21 (SE)	58°24′36″ N 15°20′57″ E	PZ, CM	FAL	510	16	44	4.6		0.06	[41]
Flakkensee Locknitz (DE)	53°26′27″ N 14°13′24″ E	CM, LV	FCPAH	550	13	21	7.1	0.27		[50]
Schuitenbeek (NL)	52°15′07″ N 5°32′20″ E	PZ	FMCFP	780	15	26	5	1.59		[50]

[a] Soils: VR— Vertisols; Fl— Fluvisols; GL, Gleysols; PZ—Podzols; AL—Alisos; AC—Acrisols; LV—Luvisols; UM—Umbrisols; CM—Cambisols. The soil classification is according to the World Reference Base for Soil Resources (this information was obtained from SoilGrids.org platform, which contains soil information on a global scale), and any adaptations from other reference systems were made using a conversion table developed by Buol et al. (2006) [51]. [b] Land use: CLDA—cereal and a low animal density; CMGFHP—cereals and mixed grassland, fruit, hops, and potatoes; CSGHDP—corn, soybeans and wheat and a high density of poultry locally; CDWLS—cereals (durum wheat, linseed, and sunflower); CDWS—cereals (durum wheat and sunflower); FAL—forest and agriculture land; FM—forest and mire; FMCFP—forests, moors, cereals, fruits, and potatoes; CPAH—cereals, potatoes, and animal husbandry; FCPAH—forest, cereals, potatoes, and animal husbandry; PPF—pasture, peatlands, and forestry; PPFAL—pasture, forestry, peatlands, and arable land; [c] reference.

2.3. Estimated P by Pedotransfer Functions

Pedotransfer functions serve to predictively extrapolate certain unmeasured soil properties using measured data from soil surveys. Pedotransfer functions that use indicators are included in the software developed to be utilised directly at the farm level by farmers, calculating the seasonal need for nutrients that could be used to reduce the use of fertilisers and thus avoid P accumulation in soils. Such software was developed to calculate the seasonal demand for P and the best cost–benefit combination of commercial fertilisers [16]. In this way, farmers should have the information necessary to apply the required doses to increase the yield of their crops, gaining benefits in both economic and environmental terms thanks to the reduction of fertilisers used and consequent P loss. However, even if P concentrations in the soil are reduced to the agronomic optimum, it is not clear whether this would be sufficient to reduce P concentrations in the runoff enough to avoid eutrophication problems [52,53]. Based on the optimal soil P threshold test (STP), which provides information on the maximum P threshold required for optimal agricultural production on farms [25], Vadas et al. [54] used the Annual Phosphorus Loss Estimator (APLE) model to predict the impact of STP reductions and erosion control measures on the transfer of soil P contents (current and drawdown) and P transport (runoff and soil erosion) in three contrasting catchment areas.

On average, each year, about 90% of P flows to rivers, lakes, oceans, or non-agricultural land, so optimising soil management and the efficient use of P would reduce nutrient pollution in intercepting waterways [55]. Overall, livestock production contributes the most to total P releases into water bodies, and the phenomenon is magnified in areas where the soils are naturally submerged or by farming practices [56].

3. Technologies to Remove P from Water

Technologies developed to remove and recover P from P-rich waste streams, such as municipal wastewaters (5–25 mg total P L^{-1}) [57], are basically of two types: physical–chemical, such as membrane filtration, precipitation, adsorption, ion exchange, or crystallisation, and biological processes (Table 3). These technologies target different P sources, using different engineering approaches that differ significantly in the P recycling rate, pollutant removal potential, product quality, environmental impact, and cost [58–60].

Table 3. Technologies to remove phosphorus from water.

Technologies	Function	Pros	Cons	Constriction	Operative Costs [a] EUR per 10^6 L of Treated Water	Cost of 1 kg of P Recovered [a]
Membrane filtration	Semi-permeable selective separation wall	Low energy cost, low capital investment, high productivity	Membrane fouling	Membrane cleaning	42–744	-
Ion exchange	Functionalised polymeric matrices	Suitable for all ions, high productivity	Economic viability	Pre-treatment	42–330	-
Precipitation	Salt added	Removal of suspended and dissolved solids	Sodium carbonate management or H_2S emissions	Plant maintenance	32–330	1.59
Crystallisation	Ca and/or Mg added	Produce granular hydroxyapatite or struvite	-	-	148–305	0.64
Coagulation/flocculation	Adding polymers or metal ions	-	-	-	32–330	-
Thermochemical treatment of sewage sludge	Mixes the ash with sodium-based salts	Produce P-enrich ash	-	Heavy metal-rich ash	28–180 *	-
Biological treatment	Selected bacteria	Low cost, high productivity	Additional treatment before P recovery	High concentrations of organic substrate	32–330	-
Adsorption	Surface phenomenon of molecular interaction	Low cost, high productivity	Reduced ability to remove organic P	Surface area and selectivity of adsorbent; contact time	42–130	-

[a] The costs expressed in different currencies were converted into EUR and discounted (http://rivaluta.istat.it:8080/Rivaluta/ accessed on 20 November 2022). * Expressed in EUR t^{-1} treated sludge.

The scarcity of raw material coupled with environmental problems related to the overuse of phosphate fertilisers has also been considered. In addition to the point sources of P, such as phosphate rocks, non-point sources containing dissolved P, such as surface water, agricultural runoff channels, or surface rainwater, from which the needed P can be drawn to sustain global needs, are considered [48,61]. Though progress has been made using strategies for the general management of fertilisation and irrigation to decrease the amounts of P losses [62], these themselves, although smaller than before, continue to reach the receiving waters [63]. However, removing P from agricultural surface wastewaters and its reuse as fertiliser could only meet one-fourth of the annual European demand and at least 3% of the global demand for P fertilisers [64,65].

3.1. P Adsorption

As mentioned above, it is important to recover P from agricultural runoff channels, but because it is not easy to intercept in that context, it is necessary to capture P directly from watercourses, where its concentration is, therefore, low. One of the most suitable techniques to recover these low concentrations of P is physicochemical adsorption, a surface molecular interaction that occurs on contact between a solid phase (the adsorbent) and a fluid (liquid or gaseous) phase (the adsorbate). The adsorption process in a solution–adsorbate system occurs because of two factors: the affinity between a solute and solvent and a higher affinity between a solute and solid. The chemical species of the adsorbates establish chemical –physical interactions through Van der Waals forces or intermolecular chemical bonds with groups of adsorbents. The adsorption process, classified among the more advanced treatments, is suitable for the removal of suspended, dissolved, and colloidal forms still present in wastewater treatment plants. The adsorption of P ions depends on different adsorbent factors, such as the surface area, charge, and physicochemical properties of the solution, P concentration, temperature, pH, and presence of other competing ions or molecules [66–68]. The selectivity of an adsorbent, i.e., its ability to remove P preferentially from competing ions, is another factor in adsorption studies and depends on the type of interaction formed by the ions competing directly for the active sites of the adsorbent surface. In general, ions such as chloride and nitrate show little or no competition, while ions such as arsenate and silicate show high competition [69–72]. Phosphate adsorption usually reaches an optimal level when the pH promotes its electrostatic attraction to the adsorbent, i.e., when the pH of the solution is lower than that of the zero-charge point (ZPC) of the adsorbent, making it electropositive. Since several adsorbents have a ZPC near-neutral pH, optimal P adsorption is often in the acidic range [73–79].

3.2. P-Adsorbent Industrial Materials: "Grey Removal"

In the beginning, the circular economy and the possibility of recycling fertiliser elements, such as P, was not a research priority given the low cost of the materials. Studies on possible biosorbents started in the 1990s, intending, essentially, to remove metal ions, organic molecules, or dyes from wastewater [80]. Since these experiments also studied the behaviours of different anions, the extension of these findings to the phosphate ion is certainly plausible. Different materials have been used for P removal from waste streams through an adsorption mechanism [81,82]. The materials most used and reported in the literature have been wastes, residues, or by-products of the metallurgical industry sometimes modified (Table 4), "grey removal". Cusack et al. [83] used adsorption to remove P from agricultural waters, employing bauxite residue, a sedimentary rock that is the source of aluminium, resulting in a potential low-cost adsorbent. They treated two freshwaters: low-P forest runoff (FR; pH 7.6 and 1 mg P L^{-1}) and high-P dairy-soiled water (DSW; pH 7.8 and 11 mg P L^{-1}). From the experiments conducted on three columns of different heights (20, 30, and 40 cm), bauxite residue had P removal capabilities in both the low and high ranges of P-concentrated waters (FR: 0.34 mg g^{-1} of P; DSW: 2.75 mg g^{-1} of P) due to the strong interaction between Al and orthophosphate anions. The estimated service times of the column media, based on the largest column studied, were 1.08 min g^{-1} for the FR and 0.28 min g^{-1} for the DSW.

Table 4. Phosphorus removal with adsorption using adsorbents derived from agricultural wastes, metal industry wastes, and by advanced biological phosphorus removal with microorganisms. The table also shows the minimum concentration of the treated aqueous solution and the pH at which the highest removal efficiency has been achieved.

Material [a]	P_0 [b] mg L^{-1}	pH	Recovery %	Ref [c]
Bauxite residue	1	7.7	95	[83]
HPMM	1	5.9	85	[77]
Zr@MCS	2	5.0	97	[84]
Chrorella vulgaris	3	7.0	93	[85]
BS	5	5.0	93	[86]
Lanthanum-based hydrogel beads	5	4.0	92	[76]
RMA	5	7.0	85	[87]
Microalgae	5	7.0	90	[88]
PAO	7	7.0	83	[89]
ZSFB	10	4.4	99	[78]
Steel chips bed	10	7.0	83	[90]
CSH	13	7.0	97	[91]
Oil shale ash	22	7.1	99	[92]
ZrMCB	40	2.0	94	[79]
JP	50	7.0	96	[93]
CH	50	7.0	83	[93]

[a] Material: HPMM—high-permeability media mixture; RMA—red mud akaganeite; ZSFB—composite fibre composed of steel slag, zeolite, fly ash, basalt; BS—sugarcane bagasse; JP—jack fruit peel; CH—corn husk; Zr@MCS—Zr immobilised on modified corn straw; ZrMCB—corn bract-modified Zr; Microalgae—*Dunaliella* sp., *Nannochloropsis* sp., and *Tetraselmis* sp.; PAO—biofilm of phosphorus accumulating organisms; CSH—hydrated calcium silicate adsorbent; [b] P_0—P initial concentration; [c] reference.

Another high-volume by-product produced in the steel industry, blast furnace slag, was used to prepare a hydrated calcium silicate adsorbent (CSH) to remove phosphate from aqueous solutions. CSH showed a maximum P adsorption capacity of 53 mg g^{-1} in a solution with an initial P concentration of 13 mg L^{-1}, at pH 7.0 and 25 °C. CSH showed excellent adsorption performance related to abundantly present Fe and Ca ions, even from phosphate solutions with a wide range of initial concentrations (2–26 mg L^{-1}) and pH conditions (pH 3–9) [91].

Sellner et al. [90] conducted laboratory experiments on fixed bed columns with recycled steel chips of different sizes using alkaline solutions. It was found that adsorption was initially rapid, followed by a stable removal step with a contact time of 3 minutes and an initial P concentration of 10 mg L^{-1} (the desorption depended on the NaOH concentration). Kasak et al. [91] treated wastewater in submerged cells with an experimental horizontal filter filled with well-mineralised oil shale ash and peat. Comparing peat and oil shale ash, the latter removed 99% of the 22 mg L^{-1} of P present in the wastewater, whereas peat removed 63%. However, oil shale ash increased the pH value and the Ca^{2+} concentration in the runoff. Ostram and Davis [77] treated simulated rainwater with a 5 cm expanded shale aggregate, based on aluminium, and a psyllium binder (*Plantago psyllium* L., a herbaceous plant that contains aucubin glycoside, polyphenols, mucilage, and other substances, with high-permeability media mixture, HPMM). P retention increased as the simulated rainfall intensity decreased, and the pH was acid. Additionally, iron oxides have been widely used; examples include fly ash and red sludge modified by $FeCl_3$ [94], where the adsorption capacity increased at pH 7. Another is akaganeite ($FeO_{0.833}(OH)_{1.167}Cl_{0.167}$) from red sludge waste, which was not affected by the pH range of the solution tested (pH 5–9) due to the zero-charge point on the sorbent and the capacity to release hydrogen and chloride ions [87].

Liu and Hu [78] used a composite fibre composed of steel slag, zeolite, fly ash, and basalt (ZSFB) in a fixed bed reactor; the best P removal occurred at pH 4 and an initial concentration of 10 mg L^{-1}. Afterwards, the fibre was regenerated with sulfuric acid. Zhou et al. [76] synthesised, characterised, and tested P removal/recovery in novel wastewater poly(vinyl alcohol)/sodium alginate/lanthanum hydroxide (PVA-SA-LH) hydrogel beads with an interpenetrating network (IPN) structure. They demonstrated a high P absorption capacity under acidic conditions due to the influence of the Lewis acid-base interactions between lanthanum hydroxide and phosphate. After five absorption–desorption cycles, the P absorption capacity of the absorbent remained above 75% of the first cycle. Many studies have tested the reusability of the adsorbent in 5 to 10 cycles.

4. P-Adsorbent Bio-Based Materials: "Green Removal"

All the studies above reported, based on the "grey" removal of P using adsorbents derived from by-products or the waste of the industries of steel, aluminium, or other material, or by their modifications, were carried out because of the availability of these materials and their chemical affinity with phosphate ions. Additionally, if often very efficient in P removal, P recovery by the adsorbent is carried out with strong acids or hydroxides. Therefore, many of these adsorbents, also meeting the criteria of a "circular economy" because the adsorbent is fully recovered and reusable several times, do not fit exactly with the concept of a "green treatment" [95]. On the other hand, relatively few works have described typical adsorption processes and the ability of adsorbents derived from agricultural waste to recover important anions such as P, arsenic (As), and chromium (Cr) (VI) anions. We identify the "green removal" agricultural waste products proposed as bio-based solutions to recover and reuse directly on farmland soils. Indeed, the feasibility of using the recovered materials in agriculture has not received much attention, however, due to their low cost, adsorbents from recovered agricultural materials deserve further study and still need major research. The great advantage in the removal of the P anions consists of the possibility, in the eventuality of a strong bind that does not allow for the recovery of P and reuse of the adsorbent, to use the product obtained as a fertiliser or a substrate. This opportunity, evidently, is not conceivable in the case of Cr or As. Table 5 lists some agricultural waste materials on which experiments have been conducted to evaluate their Cr(VI) and As(V) removal capabilities. As (V) and Cr (VI) are only considered because, in these oxidation states, they behave as anions, CrO_4^{2-} and AsO_4^{3-}, as well as P (PO_4^{3-}), and thus, these three elements behave similarly in adsorption processes. The adsorption mechanisms of other PTEs (Pb, Fe, Zn, . . .) were not considered because their behaviours are essentially those of cations.

Biosorbents tested to recover Cr(VI) work under extremely acidic pH conditions (pH 2). Therefore, the development of this solution is a challenge from an economic and chemical point of view because maintaining a pH 2 in an actual plant is far from the concept of a "low-cost and green solution". The same cannot be said for the biosorbents that have been tested to remove arsenate, which work in a pH range between 4 and 9, ensuring the promising performance of the biosorbent. Certainly, works such as those of Fox et al. [96], Gandhi et al. [97], and Asha et al. [98] will help in the development of new sustainable adsorbents for P removal, given the chemical affinity of the latter to arsenate ions.

Table 5. The adsorption capacity of biosorbents that can remove anions Cr (VI) and As (V), and the conditions under which adsorption processes occur.

Adsorbent	Modification	Pollutant Removed	Adsorption Capacity mg g^{-1}	Removal %	pH	T °C	References
Ficus auriculata leaves	Unmodified	Cr (VI)		94.3	2.0	30	[99]
Milled olive stones	Unmodified	Cr (VI)	2.3		2.0	-	[100]
Olive stone	-	Cr (VI)	53.3		2.0	30	[101]
Date pit	-	Cr (VI)	82.6		2.0	30	[101]
Cellulose derived by rice husk	Treated with alkaline humic acid	Cr (VI)	19.3		5.0	25	[102]
Exhausted coffee waste	Unmodified	Cr (VI)	686		3.0	25	[103]
Raw rice straw	Unmodified	Cr (VI)	8.0		2.0	30	[104]
Date palm trunk	Graft with diethylenetriamine and triethylamine	Cr (VI)	129.8		3.5		[105]
Sludge Biomass	Immobilised with calcium alginate	Cr (VI)	116.1		5.0	25	[106]
Sugarcane bagasse pith	Immobilised with Na-alginate	Cr (VI)	52.8		2.0	25	[107]
Black wattle tannin	Immobilised with nanocellulose	Cr (VI)	104.6		2.0	25	[108]
Cactus mucilage	Unmodified	As (V)	2.8		5.0–9.0	-	[96]
Powder of stem of *Acacia nilotica*	Unmodified	As (V)	50.8		4.0–7.0	-	[109]
Sorghum biomass	Unmodified	As (V)	2.8		5.0		[110]
Opuntia ficus indica fruit powder	Unmodified	As (V)		85–92	6.0–7.0	-	[97]
Mucilage cactus	Unmodified	As (V)		98		30	[98]
Cactus mucilage (non-gelling extract)	Mixed with sodium alginate and $CaCl_2$	As (V)	97.1				[111]
Cactus mucilage (gelling extract)	Mixed with sodium alginate and $CaCl_2$	As (V)	101.6				[111]

To take into account the environmental sustainability of the adsorbent and its usefulness and easiness for reintroducing P into the environment, over recent years, researchers have proposed some adsorbents from waste materials of the agricultural sector with good properties that would enable sustainable P recovery, both environmentally and economically. These waste materials or by-products of agricultural processing, with or without further modification, are considered environmentally friendly, low-cost, and highly selective with high adsorption capacities [112–117]. The agricultural by-products that can be used to adsorb P, and then used as fertiliser or substrate are various: apple and black currant pulp, tea scraps, banana pith, sugar cane pith, coffee pulp [118–121], orange peel, potato peel, tangerine peel, onion peel, palm peel, hazelnut peel [2,121–123], exhausted coffee, corn cobs, rice hulls, corn straw and sawdust, rice straw and husk, sugarcane bagasse [124–127], almond shells, palm shell charcoal, hazelnut shells, peanut shells, eggshell or apricot kernels, and sunflower seed shells [128–131]. Numerous attempts have been made to develop new anion exchangers by grafting positively charged amino groups onto the polymer chains of agricultural residues, such as sugar cane bagasse [86], corn bracts [93,132], raw walnut wooden shells and raw almond wooden shells [133], and wheat straw [134]. These studies have shown that the absorption capacities of the charged materials were significantly increased compared to raw materials. The reuse of agricultural waste in the form of classic fertilisers (pellets, for example) is not yet sustainable, either economically or agronomically. The main problem is the elemental composition: all the elements of plant nutrition should be present and in balanced relative quantities [135,136].

A non-alternative but potentially synergistic approach, when P concentrations are high, is to precede the bio(ad)sorption phase with growing duckweed for protein [137].

Using vegetable material (corn bracts) modified by zirconium (Zr), Jiang et al. [79] observed an adsorption rate of organic P faster than that of inorganic phosphorus. Complexation and electrostatic attraction were probably the main adsorption mechanisms. The highest removal efficiency occurred at pH 2, highlighting the limitations of this material. Another limitation of this work was the pre-treatment with Zr; the corn bract by itself cannot remove P because it lacks anion-binding sites in its original surface structure. Hu et al. [84] anchored the Zr(IV) oxide nanoparticle on maize straw grafted with quaternary ammonium groups. The resulting composite Zr@MCS showed excellent performance for P removal in terms of adsorption capacity, selectivity, and regeneration, higher than that of raw maize straw and maize bract. Again, as with the corn bract of Jiang et al. [79], the treatment of corn straw with Zr oxides made it an excellent P adsorbent but, at the same time, was a limitation for the biosorbent itself since it could not be directly used as a fertiliser or soil conditioner. The strong selectivity of the adsorbent medium was also due to the high affinity between the Zr and P, especially at an acidic pH.

Other studies have been conducted in order to increase the sustainable recovery and reuse of waste resources by evaluating the potential use of compost in combination with locally available crop residues (Table 6). This can be considered a feasible option to improve the performance of the vermicomposting process judging from the aspects of organic matter destabilisation efficiency, microbial and earthworm activity, and the fertiliser value of the final product. This can help develop alternative and environmentally friendly economic strategies for the nutrient recovery of resource-rich crop residues [122,125,138–142].

Above all, as a final consideration, with the use of materials that provide nutrients to the soil, site-specific consideration should be given to their most suitable rates and types [143].

From this analysis of P recovery solutions, it is clear that the most promising feasibility lies in direct use as a soil amendment or composting. The cost-effectiveness of P recycling, independently from the availability of a waste, depends largely on the costs of transportation, storing, and management.

Table 6. Alternative, environmentally friendly, economic strategies for nutrient recovery of resource-rich crop residues.

Production	By-Product	P Content %[a]	P Rem Min mgL^{-1} [b]	P Ads Max mg g^{-1} [c]	Use	Suitability [c]	Extra Treatment [d]	References
Banana	Fruit			1.0	Soil conditioner	+++	Compost	[138]
	Peels	0.2			Soil conditioner	+++	Compost	[139]
	Stem	0.5			Soil conditioner	+++	Vermicomposting	[140]
	Leaves	0.2			Soil conditioner	+++	Vermicomposting	[141]
	Fruit and vegetables				Soil conditioner	+++	Vermicomposting	[142]
Clover	Crop residues	0.6			Soil conditioner	+++		[144]
Hazelnut	Husk				Soil conditioner	+++	Compost	[125]
Poultry	Feather waste	0.5			Soil conditioner	+++		[145]
	Eggshell			180	Soil conditioner	+	High temp	[122]
Potato	Peel		15	190	Soil conditioner	++	Compost	[122]
Rice	Husk		1		Soil conditioner	++	High temp	[146]
		0.5			Soil conditioner	++	High temp	[146]
				12	Soil conditioner	+	Low pH	[147]
	Hull	6.0			Soil conditioner	+	Compost	[148]
Rice	Husk	0.04			Soil conditioner	+	High temp	[149]
				300	Soil conditioner	+	High pH	[127]
Almond	Shell		2		Soil conditioner	+	High pH	[126]
Sugarcane	Molasse		4.0		Soil conditioner	++		[133]
	Bagasse	0.30	10	250	Soil conditioner	++	High temp	[150]
				3.0	Soil conditioner	++	P added	[151]
	Vinasse	0.01			Irrigation	+		[152]
Sugarbeet	Root	0.5			Energy, fertiliser	+	Digestion	[153]
Orange	Peel	0.2			Fertiliser	+++		[154]
Palm	Kernel shell		1		Fertiliser	+	High temp	[123]
Peanut	Shell			30	Fertiliser	+	High temp, low pH	[131]
Coffee	Ground exhausted	0.2			Growth media	+++		[155]

[a] P content range; [b] P removal min concentration; P adsorption maxima; [c] Suitability: +++ reasonable use at farm both economically and technologically, ++ economically sustainable use at farm, + reasonable use at farm technologically only; [d] Extra treatment, at least one further treatment is required for reuse in agriculture.

5. Conclusions

Current grey P adsorbents are based on waste materials from the steel industry, which ensure a high rate of P removal but do not allow for its direct reuse as fertiliser. Green P adsorbents are vegetable wastes; they are abundant, locally available, low-cost, and eco-sustainable, but the challenge is certainly their transport. A limitation to the reuse and recycling of agricultural by-products is seeking reusability at all costs, without evaluating the technical and economic feasibility; extra interventions are frequently proposed (i.e., applying high temperatures or adding expensive synthetic molecules to modify the pH). In general, the most promising feasibility is given by its direct use as a soil conditioner or by composting it as a by-product, as the only pre-treatment.

At the time of writing, rock P costs EUR 320 per tonne, while diammonium phosphate exceeds EUR 1000 per tonne, as the fertiliser end-user price. Recovering P from surficial water can cost as much as EUR 640 per ton, depending on the type of technique used. If cheap agricultural waste adsorbents were used, the cost-effectiveness of recycling P would be even more apparent, but would also need to take into account the availability of the waste, the cost of transport, and the possible cost of storing such waste.

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