Journal of Water Process Engineering 56 (2023) 104322

Contents lists available at ScienceDirect

Journal of Water Process Engineering

journal homepage: www.elsevier.com/locate/jwpe

Drinking water treatment residuals, a low-cost and environmentally friendly adsorbent for the removal of hormones - A review

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ARTICLE INFO

Keywords:
Circular economy in the water sector
Drinking water treatment residuals
Emergent pollutants
Hormones

ABSTRACT

The declining water bodies' pristine characteristics due to the entry of emerging pollutants (EP) have been a growing concern for the past two decades. In the context of the effort that has been made to remove EP from water matrices, adsorption processes are economically attractive and feasible for EP removal. Among the commonly mentioned low-cost adsorbents (natural materials, agriculture and industrial wastes, sewage sludge or water treatment residuals), this review discusses the applicability of drinking water treatment residuals (DWTR) for the removal of hormones. DWTR have been widely reported as being effective in the adsorption of phosphate, heavy metals, and dyes. However, there is still a lack of knowledge on their application as adsorbent of hormones, such as estrone (E1), 17β-estradiol (E2) and 17α-ethinylestradiol (EE2) from water matrices. The sole study conducted on this topic, which involved a comprehensive characterization of the adsorption process for hormones using non-modified DWTR, indicates a maximum adsorption capacity of 8.748 μg/g for E2 and 14.557 μg/g for EE2. Furthermore, some studies refer to powdered activated carbon- DWTR (PAC-DWTR) as a new category of DWTR, with possible adsorption availability from powdered activated carbon (PAC) to be further explored. Finally, the application of DWTR should always be supported not only by standard toxic leaching procedures but also by ecotoxicological assessments. Nonetheless, the upcycling of DWTR into an adsorption material may offer new ways to manage this former residue in the water sector and provide alternatives for EP removal.

1. Introduction

In modern society, chemicals have become part of daily routine and are used in human activities to improve quality of life and to increase life span [1]. However, these consumption patterns are of increasing concern since these chemicals have been detected in the environment and as such may pose risks to humans and wildlife [2]. The presence of emerging pollutants (EP) in the environment has been a major concern since the 1990s [3,4]. This presence has been reported in numerous publications over the last decades in addition to research studies on their sources, fate and possible treatment solutions [5–9]. Undoubtedly, wastewater treatment plants (WWTP) have been a major point source of these compounds in the environment, as the major conventional treatment solutions are inefficient in removing them [10]. Among the wide range of treatment solutions available and described in the literature, there is a global effort for more sustainable treatment technologies [11].

1.1. The presence of hormones in the environment

Among the diverse groups of substances classified as EP, the hormone group can be highlighted, since natural (estrone and 17β-estradiol) and synthetic (17α-ethinylestradiol) hormones, are among the most potent endocrine disrupting compounds [12]. This group of compounds still does not have regulatory policies, although legal frameworks have been implemented at the European Union (EU) level, such as the Watch Lists. So far, three lists have already been published [13–15], with hormones being included in both the 2015 and 2018 Watch lists. Article 8b (2) of Directive 2008/105/EC states that the duration of a continuous watch list monitoring period for any individual substance shall not exceed four consecutive years. For this reason, hormone

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https://doi.org/10.1016/j.jwpe.2023.104322
Received 26 July 2023; Received in revised form 12 September 2023; Accepted 17 September 2023
Available online 22 September 2023
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compounds were not included in the latest watch list, published in August 2020 [15]. Given their high toxicity, the need for monitoring remains a high priority for the EU [12]. Humans and animals excrete natural steroid hormones, including estrone (E1) and 17β-estradiol (E2), through urine and feces. Additionally, the synthetic steroid hormone 17α-ethinylestradiol (EE2), which is derived from the natural estrogen E2 and used in oral contraceptives and hormone replacement therapies, take the same excretion route [17–19]. Table 1 presents the main chemical and physical properties of E1, E2 and EE2. Estrogens belong to tetracyclic aliphatic hydrocarbon compounds, consisting of one benzene ring, two hexadecane and a five-carbon ring and the major structural difference between E1, E2 and EE2 lies on the carbon bond C_{17} [20]. The acid dissociation constants, pKa, for all three compounds exceed 10, indicating that they predominantly exist in their molecular forms under both acidic and neutral conditions. Furthermore, their high octanol-water partition coefficients, Log K_{ow}, underscore their significant hydrophobicity and together with the limited water solubility, collectively contribute to their long-lasting presence in aquatic environments [20,21].

Natural steroid hormones (endogenous estrogens) have higher estrogenic potency compared to exogenous estrogens, so there is a direct relationship between the increasing global population and increasing estrogenic contamination of the environment [12]. This group is categorized as endocrine disrupting compounds (EDCs), known for their ability to alter the natural functioning of the endocrine system in wildlife. They achieve this by either blocking or mimicking the normal actions of endogenous hormones, impacting hormone synthesis or metabolism, and causing disturbances in hormone levels [17,22–25]. Furthermore, these chemicals have the potential to enter and biomagnification throughout the food chain, posing risks to ecosystems, including aquatic biota [9,17,19]. Dang and Kienzler [26] conducted a comprehensive review on the widespread feminization of male fish. Their analysis of sex ratio data for zebrafish (*Danio rerio*), medaka (*Oryzias latipes*), and fathead minnow (*Pimephales promelas*) revealed that approximately two-thirds of the studies demonstrated fish feminization following exposure to E1, E2, and EE2.

The assessment of environmental risks associated with E1, E2, and EE2 has been traditionally conducted by calculating a risk quotient (RQ), which represents the ratio between the measured environmental concentrations (MEC) and the predicted no-effect concentration (PNEC). Based on this approach, RQ values can be categorized as negligible risk ($RQ < 0.01$), low risk ($0.01 > RQ < 0.1$), medium risk ($0.1 > RQ < 1$), and high risk ($RQ > 1$) [29]. Studies on the ecotoxicological risk assessment of single exposure to E1, E2 and EE2 have always referred to these compounds as high-risk substances sampled in rivers and WWTP, with RQ values always >10 [30–32]. In a study conducted by Riva et al. [32], RQ values exceeding 150 were observed for E2 and E1. Despite the fact that the predicted and measured concentrations of estrogens in the study's receiving streams were deemed very low, the inherent high toxicity of these substances led to low PNEC values, thereby resulting in elevated RQ values. When evaluating all examined water bodies, the compounds were ranked in the following order based on their environmental risk: EE2 > E2 > E1 [33].

1.2. Hormones walkthrough in a conventional wastewater treatment plant

Conventional WWTP were not designed to remove EP, with the aggravating factor that these compounds are generally detected at low concentrations, ranging from ng/L to mg/L [9,23,34,35]. As a result, non-degradable and non-removed substances have been detected in WWTP discharges and surface waters receiving such discharges [1,6,7,36]. The presence of E1, E2 and EE2 has also been reported in sewage sludge and biosolids WWTP, due to their adsorptive character [19]. Agricultural reuse of WWTP sludge as fertilizer is a common practice as it provides nutrients and gives structure to the soil. Nevertheless, sewage sludge, with its intricate composition comprising organic matter, nutrients, and detrimental elements like heavy metals, organic micro-pollutants, and pathogens, can potentially serve as a conduit for contamination [9,37]. As such, both liquid and solid treatment phases in a conventional WWTP are pathways for EP to enter the environment [9]. Based on the systematic review conducted by Tiedeken et al. [8], on the presence of E2 and EE2 in wastewater treatment plant (WWTP) discharges and receiving waters, it was found that the concentrations of E2 and EE2 in surface water generally remain below 50 ng/L and 10 ng/L respectively. However, these values surpass the recommended annual average environmental quality thresholds for these substances (0.04 ng/L and 0.035 ng/L respectively) established in the EU watch lists of 2015 and 2018 [13,14]. There is scientific consensus that WWTP and livestock wastes are the main cause of steroid contamination in the environment worldwide [2,12,33]. It is noteworthy that WWTP are not effective in removing steroids throughout the treatment process [23,25]. Recently published reviews at different geographical scales have compiled the range of concentrations in which these compounds are detected in WWTP influents and effluents, as presented in Fig. 1 and detailed in the supplementary material (Table S1).

During conventional wastewater treatment, the removal rate of hormones depends primarily on biodegradation and adsorption processes. In essence, hormone adsorption in the wastewater treatment system relies on the transfer of mass from the aqueous phase to the corresponding solid phases. It has been reported that sludge treatment (both primary and biological sludge) can retain about 34 %–100 % of steroids [19]. The log K_{ow} values for estrogenic compounds, varying between 2.8 and 4.2, indicate that hormones are moderately hydrophobic organic compounds, showing that they have low solubility in water and favouring adsorption onto solids [17,19,38,39]. The processes of hormones biodegradation by microorganisms can occur in three different ways, namely by using the carbons of the substance as an energy source, the use of enzymes (produced by microorganisms) capable to biotransform them into different products and converting steroids

Table 1
Chemical and physical properties of estrone (E1), 17β-estradiol (E2) and 17α-ethinylestradiol (EE2).

Name of substance	CAS	Molecular formula[a]	pKa	Log K_{ow}[a]	Henry's Law constant	Solubility in water (mg/L)	Molecular weight[a] (g/mol)
Synthetic hormones							
17α-Ethinylestradiol (EE2)	57-63-6	$C_{20}H_{24}O_2$	10.33[b]	3.67	7.94×10^{-12} atm m/mol at 25 °C[a]	11.3 at 27 °C[a]	296.4
Natural hormones							
17β-Estradiol (E2)	50-28-2	$C_{18}H_{24}O_2$	10.46[a,b]	4.01	3.64×10^{-11} atm m/mol at 25 °C[a]	3.90 at 27 °C[a]	272.4
Estrone (E1)	53-16-7	$C_{18}H_{22}O_2$	10.33[b]	3.13	3.8×10^{-10} atm m/mol at 25 °C[a]	30 at 25 °C	270.4

[a] [27].
[b] [28].

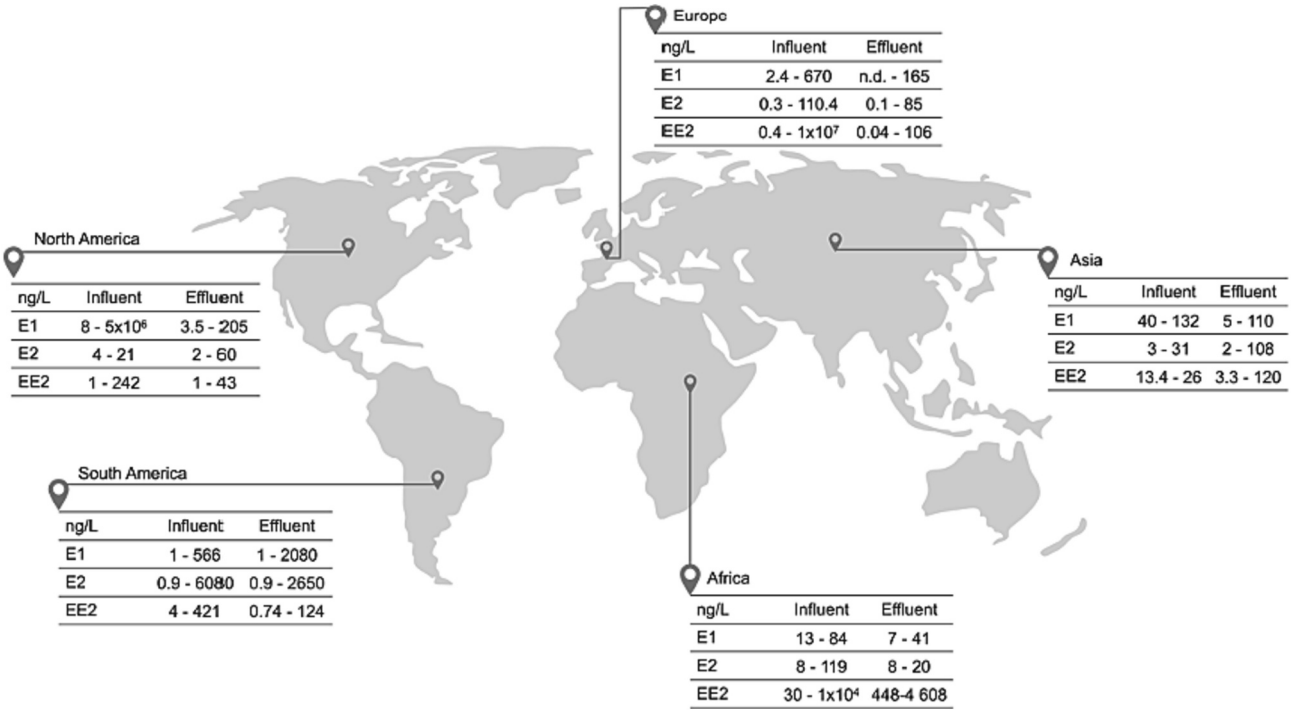

Fig. 1. Concentrations of estrone (E1), 17-estradiol (E2) and 17-ethinylestradiol (EE2) reported in WWTPs influents and effluents in different continents.

into metabolites, although, the latter are difficult to be degraded by these microorganisms [39].

1.3. Drinking water treatment residuals - a low-cost adsorbent

Extensive research has been conducted in the past decades to explore advancements in wastewater treatment processes. The primary objective has been to reduce the release of EP into receiving waters while enhancing the overall quality of effluents for potential water reuse [40]. The objective of upgrading and implementing design improvements in conventional WWTP is to convert EP into compounds that are less harmful or even eliminate them entirely [41]. New fastest-performing technologies in wastewater treatment include i) physical methods such as adsorption (through adsorbents such as activated carbon, biochar, carbon nanotubes, and clay minerals), and membrane systems, ii) chemical methods such as advanced oxidation processes (AOPs) and ozonation [6,42–44]. Various chemical processes, including electrochemical oxidation, photo-electrochemical oxidation, Fenton oxidation, photo-Fenton oxidation, UV/TiO2, photocatalytic ozonation, and hybrid processes, have been extensively studied for EP removal. However, their full-scale application as advanced treatment methods for urban wastewater is hindered by technological limitations and high costs. Consequently, these processes will not be discussed in the current review [10,44,45]. Among advanced wastewater treatment, ozonation, adsorption processes (mainly using activated carbon) and membrane separation are commonly referred to as conventional techniques. There is scientific consensus among these that adsorption processes are one of the friendliest, considering operational management, the formation of by-products during the process and the quality of the final effluent. Even though there has been a struggle to search for new reusable raw materials, low-cost adsorbents, instead of the conventional mineral coals, from non-renewable sources, that are commonly used in the production of activated carbon [46,47].

Drinking Water Treatment Residues (DWTR) constitute a by-product formed during the process of drinking water treatment, and their prominence has increased due to a trend in production growth [48]. This by-product emerges during the treatment of drinking water and primarily comprises aluminium or iron salts. These salts are introduced during the coagulation phase to promote the aggregation of particles in the untreated water, facilitating subsequent sedimentation [49]. The composition of DWTR is closely linked to the chemical characteristics of the raw water source (whether it is derived from surface water or groundwater) as well as its geographical location [49–51]. These factors exert an influence on the chemicals employed during water treatment, namely Al and Fe salts, oxidizing and pH adjusting agents and flocculants like cationic or anionic polyelectrolytes, which are added to adhere to regulatory requirements for potable water. Beyond the chemicals introduced during treatment, the composition of DWTR can encompass suspended particles, organic matter, and dissolved ions such as Ca^{2+}, Fe^{2+}, Mn^{2+}, and humic acids derived from the composition of the untreated water [49–52].

It is estimated that the proportion of generated DWTR is 1–3 % of treated water (measured in m^3) in a drinking water treatment plant (DWTP) [53,54], corresponding to the generation of millions of tons of DWTRs every day, globally [51]. These residues are normally sent to landfill disposal, since their composition is mainly chemical and, therefore, their direct application to soil may pose environmental risks due to the possibility of aluminium leaching and the presence of other chemicals [54,55]. Despite the ongoing debate regarding the benefits of using this material in agricultural applications, there has been a recognition of its potential for soil pH amendment, soil conditioning, and remediation. These positive prospects offer a pathway to reclaim problematic and unproductive soils, ultimately improving agricultural productivity [56]. Nevertheless, it has been documented that these residues are suitable for reuse in various other endeavours. They can be employed as aggregates in the construction sector, utilized to produce environmental remediation materials, and even serve as coagulants in wastewater treatment processes [48,54,57,58]. Within the water sector, DWTR are suggested as a suitable low-cost adsorbent material as a substitute for conventional activated carbons to be used in water matrices treatment, such as wastewater [53,57,58].

When associating DWTR with the challenge of eliminating EPs from water matrices, a notable knowledge gap became apparent. This void stems from the insufficient information regarding the feasibility of

employing this material to adsorb such compounds. While the principal objective of this study is to systematically review the implementation of DWTR in predominantly targeting hormones like E1, E2, and EE2 within water matrices, along with an investigation into the fundamental mechanisms governing the adsorption process of these substances, it will additionally explore the effectiveness of DWTR in adsorbing other EPs.

Several other reviews related to the application of DWTR for the removal of EPs from water matrices have been published. These include i) a review focusing on its use in stormwater as a filter medium within bioretention systems, serving to adsorb phosphorus and also acting as a pathogenic retention system [59]; ii) three reviews addressing the valorisation of DWTR, which outline its adsorption capabilities for heavy metals, dyes, and phosphorus [49,53,60]; iii) an examination of the circular economy aspects concerning the reuse of DWTR [51], which underscores its adsorption capacity for various pollutants such as fluoride, perchlorate, hydrogen sulfide, sulfur dioxide, and carbon dioxide; and iv) an recent review on several types of adsorbents performance in the removal of E1, E2 and EE2, though it does not include DWTR in the types of material studied [20].

A common conclusion in these reviews is the acknowledgment of DWTR's potential as a viable adsorbent. However, it is noteworthy that there are no existing reviews specifically addressing the adsorption of hormones.

2. Methods

The aim of this study was to review the scientific literature published in the last two decades addressing the use of DWTR (with no activation/reactivation process) directly on water matrices to remove steroid hormones. For this purpose, the literature review was structured based on the approach proposed by the PRISMA statement [213], to ensure data quality. This process involves several steps, including establishing search parameters (such as databases, search times, and publication types), selecting appropriate search terms, formulating eligibility criteria for inclusion and exclusion, conducting the literature search, and undertaking the review and selection of articles. These steps culminate in the creation of a comprehensive database of publications and the subsequent analysis of the literature (Table 2).

Through the raw data retrieved from the Scopus database, using the search Q presented in Table 2, it is possible to see a growing scientific interest in the use of DWTR, linked to the adsorption process (Fig. 2).

In addition, the same search results were analysed using the VOSviewer software, resulting in a keyword density network between the retrieved documents that identify the relatable subjects, as shown in Fig. 3. The larger the circles, the higher the density of a specific keyword.

After identifying all potentially relevant articles, a selection process was carried out to find articles for inclusion that met the eligibility criteria, by title and abstract analysis, as presented in Fig. 4.

Within the document pool, 46 documents were selected, corresponding to those strictly related to the application of DWTR in water matrices to remove one of the selected compounds or the entire group, or even other pollutants. The remaining 78 documents used in this section were selected due to what may be called a "snowball effect", leading to an increasing number of relevant papers in the selected chapter (through reference lists and papers citing key papers found) that were not identified by the selected keywords or in Scopus and Web of Science databases. Moreover, the extra documents identified were not always strictly related to the application in the search Q but were complementary for the information structure accuracy. A total of 124 documents were selected to be used in the next section.

Table 2
Eligibility criteria used for literature review (for title and abstract filter).

Research question	
Are drinking water treatment residuals (DWTR) a possible material to be reused for the adsorption of hormones and other pharmaceuticals?	
Eligibility criteria	
– Specifically, discuss at least one of the three compounds of interest;	
– May include other types of compounds which reinforce the applicability of DWTR, such as antibiotics, pesticides, phosphorous, heavy metals and dyes;	
– DWTR must only be thermally modified, air-dried or in a raw state;	
– The applicability of the DWTR must be in water matrices;	
– Must be a peer-reviewed original article, review or book chapter;	
– Only English-written documents;	
– Includes peer-reviewed original articles referenced by the selected documents;	
– Full text must be available	
– Must be published between 2000 – September 2023 (updated in February 2023)	
Search Q	(TITLE-ABS-KEY ("water treatment residuals") OR TITLE-ABS-KEY ("water treatment sludge") OR TITLE-ABS-KEY ("drinking water treatment sludge") OR TITLE-ABS-KEY ("drinking water treatment residuals") OR TITLE-ABS-KEY ("waterworks sludge") OR TITLE-ABS-KEY ("waterworks residuals") AND TITLE-ABS-KEY ("adsorption")) AND PUBYEAR > 1999 AND (LIMIT-TO (LANGUAGE, "English")) AND (LIMIT-TO (DOCTYPE, "ar") OR LIMIT-TO (DOCTYPE, "re") OR LIMIT-TO (DOCTYPE, "ch"))

3. Results and discussion

Undoubtedly, the range of advanced wastewater treatment options is vast, and it is difficult to identify the best solution to reduce EP entry into the aquatic environment. This decision should be based on the specific objectives for each implementation project, which depends on local conditions, such as space availability, energy costs, capital availability and quality of the required effluent [61]. However, there is a general scientific consensus on the use of technologies based on adsorption processes [10,11,44], as they offer the most attractive set of factors as they are technologically simple and efficient, can be integrated with other technologies (hybrid systems), allow low-cost treatment, do not produce toxic by-products during the process and can integrate other sectors waste as raw material to produce activated carbon, reducing its carbon footprint and therefore a more sustainable solution [45,62]. In Europe, the activated carbon commonly used for water and wastewater treatment installations is produced from bituminous coals or coconut shells and both these sources have environmental constraints. Coal extraction is related to serious environmental and social impacts, which are characteristic of non-renewable resources. The utilization of coconut shells, on the other hand, promotes the development of monocultures, which has adverse effects on land use. Additionally, it entails the use of chemical fertilizers and pesticides, potentially compromising the quality of water bodies [62]. The largest production of these raw materials occurs in Asian countries, which implies high transportation costs and a higher carbon footprint. The use of local raw materials from different economic activities, such as agriculture or industrial wastes, is a logical way to overcome the environmental constraints mentioned above. The significance of these waste types lies in their contribution to achieving a circular economy and promoting sustainability. Prioritizing their prevention, reuse, and recycling within the waste management cycle is crucial for realizing these goals [63,64]. According to Singh et al. [65], adsorbents for EP removal can be divided into five different categories, i) activated carbon adsorbents; ii) non-conventional low-cost adsorbents; iii) nano-material adsorbents; iv) composite and nanocomposite adsorbents and v) a category of miscellaneous adsorbents. As the most sustainable adsorption option, the category of non-conventional low-cost adsorbents comprises the most suitable raw materials for this purpose. This category includes adsorbents such as natural materials (i.e. wood, coal, peat moss, chitin/chitosan, clays and natural zeolites), agriculture wastes (i.e. vegetable and fruit peels, wheat bran, rice husk, coconut shells and pulse seed coat), industrial wastes (i.e. fly ash, palm oil ash, red mud, bagasse ash and coffee waste) [45,66–69] and sewage sludge or water treatment residuals [53,55,70,71].

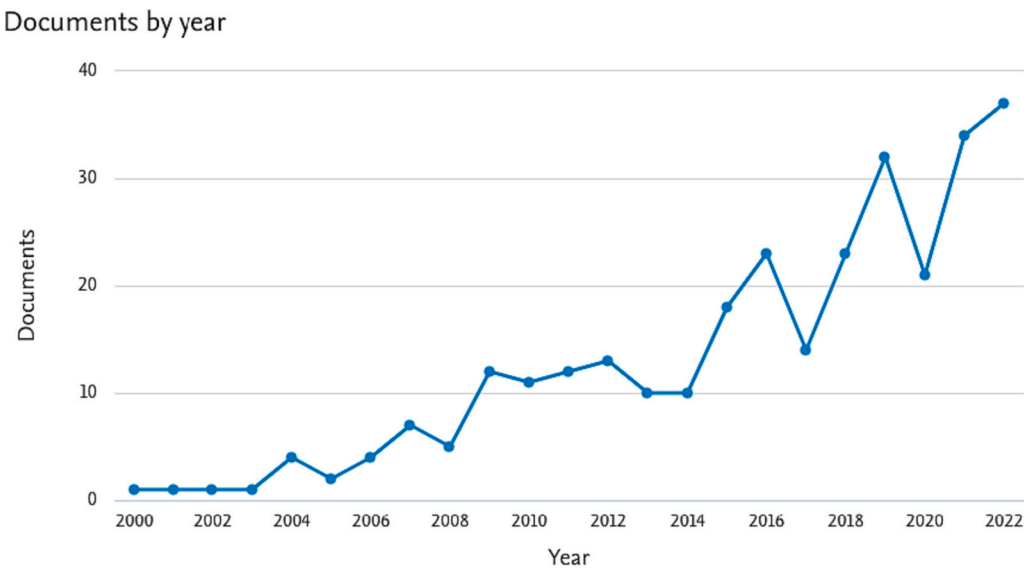

Fig. 2. Results of the Scopus Q survey, by year (retrieved from the Scopus database).

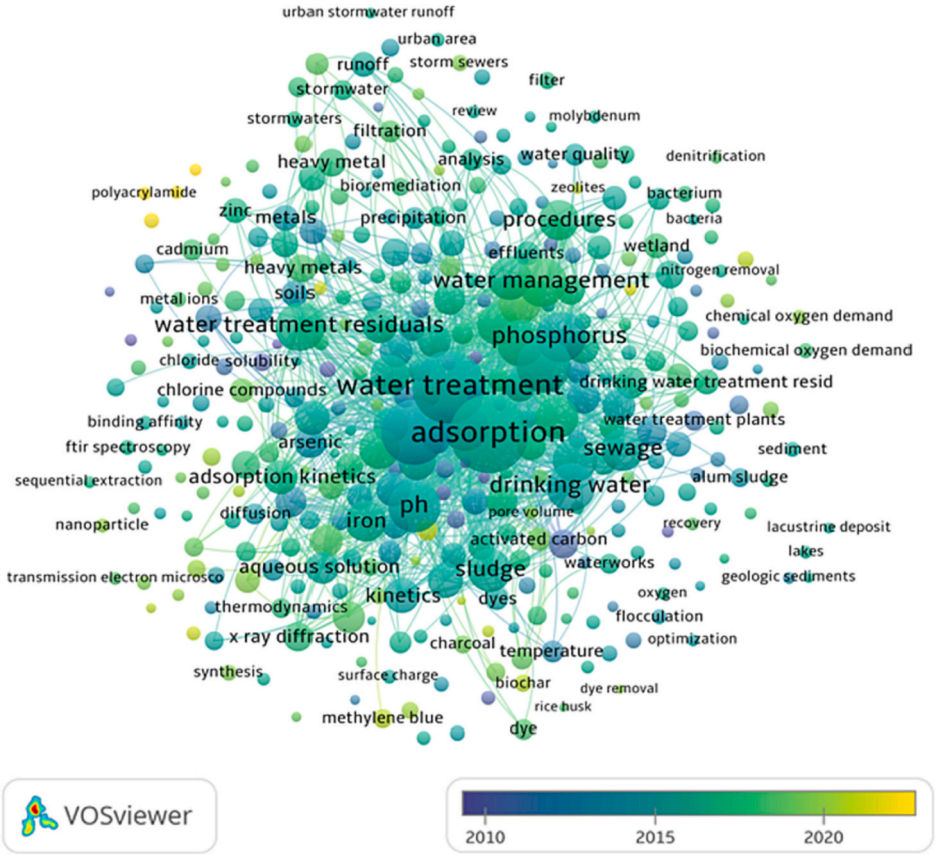

Fig. 3. VOSviewer network analysis.

3.1. Drinking water treatment residuals for a more sustainable advanced wastewater treatment

Regarding the water sector, particularly DWTPs, there has been an increasing effort to provide access to clean water globally, as it is estimated that one in three people worldwide still lack access to safe drinking water [72,73]. Such increasing consumption has a direct impact on by-products/waste generation, namely DWTR. These are a direct and unavoidable result of the drinking water treatment process, in which coagulants and powdered activated carbon (PAC) are, frequently, used for impurities aggregation and settling and toxins adsorption, such as pesticides and cyanotoxins but also taste and odour removal of the raw water, respectively [54,74].

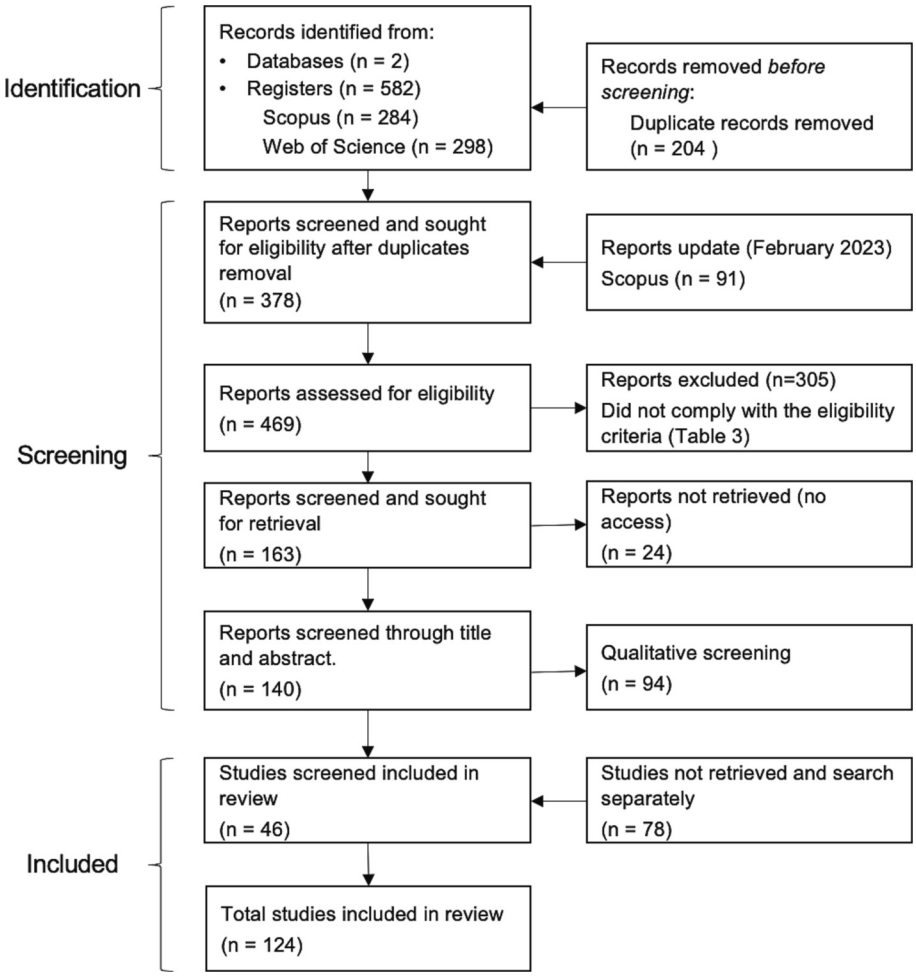

Fig. 4. Methodological approach.

3.2. Alternative disposal and possible applications of DWTR

The disposal of DWTR has always been a problematic and costly operation for water management entities since the common methods for this purpose are getting limited. Landfilling remains one of the most common final disposal destinations although it is an unsustainable practice per se [55]. Furthermore, land application of DWTR for agriculture purposes is quite limited since there is concern regarding the toxicity and metal content of DWTR leaching into the soil [48]. Within the "closing the loop" concept, proposed in the Circular Economy Plan by the European Commission [75], the reuse of DWTR is a step closer to reaching a full circularity in the water sector. Several valorisation procedures have previously been reported and include: i) a reactive fill media for constructed wetlands, reed beds and other types of filter beds [76–80] ii) as part of constructive materials [81–85], iii) for coagulant recovery and reuse [86–89], iv) some special land uses [90–93] and finally v) as a low-cost adsorbent material [94–96].

3.3. DWTR physicochemical characterization and adsorption process

DWTR are produced daily worldwide, resulting in thousands of tonnes of this material being available for potential reuse. Before considering its potential application, one may choose to apply modification methods, such as regeneration steps (though not strictly mandatory), followed by necessary characterization steps. These characterization steps are vital for understanding the physicochemical properties of the adsorbent material.

3.3.1. DWTR characterization

Before delving into the modification treatments for DWTR, there are standard pre-treatment procedures. These typically involve dewatering of DWTR, often conducted locally at the DWTP, followed by air drying or low temperature drying, and ultimately crushing and sieving the material [53,97]. Several modification methods can be applied to enhance the adsorption performance of DWTR (as outlined in Table 3). These modification approaches encompass thermal treatment [74], chemical treatments under acidic or alkaline conditions, physical surface modification via surface coating, improvement/amendment/compositing with industrial or agricultural waste materials, nanoparticle synthesis, and granulation [97].

Indeed, modification procedures alter the morphological and physicochemical characteristics of DWTR, typically for the better. Nevertheless, it is important to note that they come with substantial costs [51,97]. Hence, when considering the modification of DWTR for reuse applications, careful consideration is necessary. Some reuse purposes demand minimal material processing, as the raw material itself is suitable. Examples include its use in landfill lining or capping, agricultural applications, or as a substrate or filter media in wastewater treatment plants [51].

In the context of this review, we present and characterize DWTR as a ready-to-use raw material for reuse applications. Therefore, further characterization of modified DWTR was not explored.

The characterization of DWTR is obtained through several different types of analysis, such as elemental composition which is used to quantify the carbon, hydrogen, nitrogen and sulfur content. Proximate analysis provides information on moisture content, volatile matter and

Table 3
Modification methods for DWTR.

Type of modification	Treatment characteristics	References
Thermal treatment	DWTR are submitted to high temperatures to eliminate the excess of organic matter (OM) naturally present in the material which occupy available adsorption sites. An optimum temperature is crucial to prevent adsorption capacity loss, since OM also contribute for adsorption of hydrophobic compounds and the excess of temperature leads to material crystallization, reducing, adsorption capacity. Most common thermal treatments include pyrolysis (biochar production), calcination and thermal roasting.	[7497,98]
Chemical treatment	Main purpose is to change the surface functional groups and charge to promote the protonation or deprotonation of actives sites.	[97]
Acid conditions	DWTR are submitted to a water-washed followed by acid washed solution. The high acid content will change the surface morphology. Also, it will promote OM volatilization, reducing carbon content and thus increasing the ash content. These acid conditions will protonate the DWTR surface and promote the adsorption of anionic contaminants.	[97,99,100]
Alkaline conditions	DWTR are washed with an alkaline solution with functional groups enriched with oxygen, modifying the surface charge by increasing negative ions in it. Therefore, the active sites will be deprotonated and will promote the adsorption of cationic contaminants.	[97,101]
Surface modification	DWTR are added/loaded with several metals like Fe, Cu, Pt, Ag, and La to enhance its surface characteristics, such as surface area and pore volume. These modifications are especially effective up to a certain optimum metal loading, which greatly improves the adsorption capacity for pollutants.	[97,102,103]
Compositing	Combining DWTR with other materials to enhance their physical and chemical properties that will ultimately in higher adsorption capacity. Several materials can be used for this purpose such as natural residues (e.g. wood mulches) and industrial residues (e.g. tire rubber).	[97,104,105]
Nanoparticles synthesis	Reducing the particle size of DWTR bellow 100 nm, stabilizing the material and which will increase the surface area and pore volume by 2–3 times and actives sites up to 30 times compared to powdered raw DWTR.	[97,106,107]
Granulation	To address issues related to the material stability of DWTR, such as physical particle disintegration, granulation emerges as a promising modification technique. This process involves transforming DWTR, whether it is in powdered, fine, or coarse form, into a granulated, pelleted, or beaded format through aggregation. The primary goal of this modification is to enhance hydraulic characteristics, particularly by boosting hydraulic conductivity and compressive strength when DWTR is employed in filter media. Additionally, granulated DWTR offers practical benefits, including ease of transportation and straightforward separation and recovery from water after adsorption. These advantages make granulated DWTR exceptionally well-suited for use in wastewater treatment plants, where they can be effectively utilized in columns, beds, and filters.	[97,100,108–112]

ashes quantification. The correlation between the elemental analysis data and the ash content allows the quantification of the oxygen content in the material [94].

Within the scope of the characterization of DWTR, mineral content analysis is the quantification of selected elements, as Ca, K, Na, Fe, Mg, Al, Cr, Ni, Cu, Zn, and Si. DWTR are extremely rich in amorphous Fe or Al oxides, due to the use of Fe/Al coagulants in the water treatment process [53,59]. Dias et al. [94], tested two different DWTR (different sources) for the adsorption of estrogens, both with PAC incorporation, and their element composition showed the same proportion of Al content, as the major element. However, the incorporation of limestone insoluble in one of them increased the Ca^{2+} content. The mineral content analysis can also provide information on the feasibility of land applications [53]. Textural parameters are presented as part of the material characterization, giving information on the specific surface area, pore volume, and area, as well as the pore size distribution of the material, using Brunauer, Emmett, and Teller (BET) model, which may lead to a first insight on the adsorption performance [113]. Each of these parameters plays an important role in the adsorption process. For example, surface area measures the total area available on both the external and internal surfaces of the adsorbent for adsorption. A larger surface area results in more contact points for adsorbates, leading to higher adsorption capacity and faster adsorption kinetics. This surface area is composed of pores with varying sizes, ranging from micro to macro porosity. Greater pore volume equates to more space for adsorbates, a crucial factor, especially for larger molecules or ions [113]. Active sites, serving as the locations where interactions between the adsorbent and adsorbate molecules occur, are essentially binding sites. Consequently, a higher count of active sites leads to heightened interactions and, consequently, greater adsorption capacity [114].

Table 4 presents examples of the composition of two common types of sludge described in the literature, aluminium-DWTR (Al-DWTR) and ferrous-DWTR (Fe-DWTR), and a third recently described as a DWTR with powdered activated carbon (PAC-DWTR) [74,115]. In warmer weather conditions, the most recent classification, PAC-DWTR, involves the inclusion of PAC before adding aluminium in the DWTP. This step is taken to enhance the adsorption of organic compounds, which can potentially result in taste and odour issues, as well as to capture cyanotoxins, and to counteract contaminants stemming from the seasonal runoff of agricultural chemicals [115]. Due to the escalating pollution of raw water sources and the need to adhere to legal requirements for drinking water treatment, there has been a notable upsurge in the use of PAC [74]. In fact, the use of PAC has also already been a common practice in Europe and the United States for several years [116]. Consequently, the production of PAC-DWTR is becoming increasingly prominent. Using PAC-DWTR offers distinct advantages over conventional Al/Fe-based DWTR. This material not only incorporates Al/Fe salts into its composition (depending on the coagulant used during water treatment), but it also boosts a higher carbon content due to the presence of PAC. This heightened carbon content enhances the material's adsorption properties, as exemplified in the study conducted by Dias et al. [94]. Additionally, the feasibility of thermally reactivating this material without causing environmental harm has been demonstrated by Lee et al. [74].

Even though, it must be always take into account that the specific physicochemical properties of DWTR are closely related to the

Table 4
Example of possible composition of three different types of drinking water treatment residuals (DWTR).

Parameters	Al-DWTR[a]	Fe-DWTR[a]	PAC-DWTR[b]
pH_{pzc}	6.85 ± 0.13[c]	7.50 ± 0.11[c]	7.46 and 11.29[d]
Total pore volume (cm^3/g)			0.024, 0.065 and 0.161
Surface area (m^2/g)	134,1 ± 131,5	28[c]	6.54, 127[c] and 318[c]
Mineral analysis (mg/kg)			
Al	118,700 ± 24,260	61,390 ± 35,920	121,383 ± 117,190
Ca	10,360 ± 4299	n.d.	43,927 ± 61,880
Cd	0.12 ± 0.02	n.d.	n.d.
Cr	20 ± 7	38 ± 4	n.d.
Cu	624 ± 581	46 ± 12	7 ± 5
Fe	37,000 ± 19,740	220,900 ± 32,200	18,004 ± 17,156
Hg	0.46	n.d.	n.d.
K	3547 ± 582	n.d.	2465 ± 3928
Mg	2407 ± 572	n.d.	3335 ± 1809
Mn	2998 ± 1122	1088 ± 178	2473 ± 3751
Na	355 ± 142	n.d.	174 ± 283
Ni	28 ± 10	64 ± 14	n.d.
P	2030 ± 1070	710 ± 220	24,288 ± 17,492
Pb	22 ± 12	47 ± 1	14 ± 1
Zn	98 ± 31	36 ± 4	19 ± 4
S	6763 ± 2955	n.d.	2333 ± 1101

[a] Based on [48,19], inputs from [52,59,87,122–127].
[b] Inputs from [74,94].
[c] [128].
[d] [94].
[e] Single point method at the relative pressure of $p/p_0 = 0.3$.

characteristics of the raw water to be treated, the coagulant choice and the type of treatment, therefore, these characteristics are extremely variable [48,52,53,87], and sometimes can be visible (Fig. 5).

Solution pH is of utmost importance in the adsorption process to assess the behaviour of the adsorbent material in a specific solution. To this end, the pH analysis at the point of zero charge (pHpzc) of the material will determine the pH at which the adsorbent surface remains neutral [117]. Hence, this parameter enables the prediction of surface functional group ionization, facilitating the evaluation of interactions between the adsorbent and adsorbate. When the solution pH exceeds the pHpzc (point of zero charge), the adsorbent surface carries a negative charge and exhibits an affinity for cations (positively charged species). Conversely, when the solution pH is lower than the pHpzc, the surface becomes positively charged, promoting interactions with negatively charged species (anions) [118,119]. Regarding E1, E2 and EE2, and recalling their pKa above 10, it is expected that in alkaline conditions, electrostatic repulsion is formed between estrogens and the adsorbent

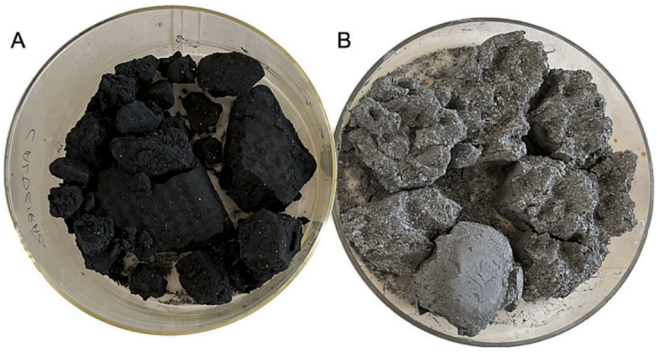

Fig. 5. Two different types of drinking water treatment residuals (DWTR); A) raw PAC-DWTR without sieving; B) raw Al-DWTR with high content of insoluble limestone, without sieving.

[20], which is a drawback when using chemical reactivated adsorbents in alkali conditions, such as KOH-activated biochars [120].

For instance, Martins et al. [117], who have studied the adsorption of estrogens using DWTR, due to the anionic characteristics of the material, pHpzc of 6, choose to lower the pH of the solution to 5.5, to increase the electrostatic interaction between adsorbent and adsorbate. Therefore, within this process, the pKa of the molecule of interest must also be considered to ensure its full solubility [117].

To predict and understand the thermal behaviour of the DWTR when exposed to a physical regeneration or activation process, thermogravimetric analysis (TGA) provides information on the material decomposition with increasing heating rate but also changes in molecular structure [117]. The first endothermic process within TGA is normally related to water losses (evaporation), followed by organic matter volatilization and finally structural modifications due to the breakdown of stronger chemical bonds [94,117,121]. Within the application of DWTR for the adsorption of hormones, Dias et al. [94] reported a total mass loss of 34 % and 27 % in each DWTR tested, while Martins et al. [117] reported a 21 % loss. Ideally, the lower the total mass loss, the more stable the material will be and with a greater possibility of successive regenerations.

Scanning Electron Microscopy (SEM) is an analytical technique used to study surface characteristics, particle arrangement and changes which may have occurred after adsorption processes [129,130]. DWTR in their raw state tend to show a perceptible heterogenous structure, which, according to Martins et al. [117], tend to get a smoother and smaller particle size after thermal and chemical modification. Energy Dispersive Spectroscopy (EDS) is a technique normally coupled to SEM in which the type of elements in the adsorbent material sample, are identified and their relative ratios are quantified, supporting the elemental analysis [129,131,132].

Surface functional groups are determined through Fourier transform infrared spectroscopy (FTIR), where each inverted peak on the graph corresponds to a specific chemical bond. This analytical method is employed to examine alterations in functional groups, particularly those involving π–π interactions and hydrogen bonding, by comparing the spectroscopy of adsorbents before and after adsorption [20]. Also, FTIR information may provide insights on the adsorption mechanism that is responsible for the adsorption process. DWTR are heterogeneous and its composition is variable, as mentioned above, and therefore different functional groups are expected on the surface. However, several researchers have identified the O—H stretching related to hydroxy group to be between 3570 and 3200 cm^{-1}, a C═C double bond of aromatic organic matter around 1600 cm^{-1}, and the presence of mineral functional groups (e.g. Al-OH, Fe-OH or Fe—O) due to the presence of Al and Fe oxides, but also Si related stretching bonding's with those oxides [99,117,130,131,133]. The only conducted study into the adsorption of E2 and EE2 onto DWTR [117] did not assess changes in the stretches identified in FTIR analysis after adsorption. However, other researchers who focused on the adsorption of estrogens onto different types of adsorbents have identified modifications in C═C stretches, including a decrease in peak intensity or even their disappearance. These observations suggest that π-π interactions play a significant role in the adsorption of estrogens [20,134,135]. Also, O—H stretching shifting from around 3400 to 1370 cm^{-1} have been identified, indicating formation of hydrogen-bounding interaction [20,135–137].

3.3.2. DWTR hormones adsorption process

3.3.2.1. Adsorption dosage. Concerning modelling the adsorption of specific compounds, dosage studies are typically used to assess the feasible dosage necessary to adsorb each target compound. Concerning its procedure, it works by fixing the target compound concentration and contact time and varying the adsorbent dose [130]. The outcome of this analysis is the adsorption capacity (q_e), which commonly increases as

the adsorbent dose decreases. This could be explained by mass transference phenomena, where a lower mass dosage allows all active sites to be readily available for surface adsorption and thus be rapidly saturated, increasing the adsorption capacity [117,118,129]. The opposite, i.e. the decrease in q_e values, is usually observed as the adsorbent dosage increases, which can be related to the lower number of active sites due to particle aggregation [129]. According to Martins et al. [117], the adsorbent dosage that reach the highest adsorption capacity for the removal of E2 and EE2 was 0.5 g (10 mg/mL) at an initial hormone concentration of 100 μg/L.

3.3.2.2. Adsorption kinetics. Adsorption kinetic studies are used to assess the transference rate of the adsorbate into the adsorbent, to evaluate the performance of the adsorbent material and to determine mass transference mechanisms. The kinetic mass transference data will directly affect the adsorption design system since it provides information on the maximum contact time for full adsorbent saturation, hence it is a crucial step [138]. There are several kinetic models used to describe the adsorption process, the classic approaches use the pseudo-first order (PFO) and pseudo-second order (PSO) rate equations [139], but also Elovich, Avrami, Crank, Vermeulen, Weber-Morris, Bangham, linearfilm, mixed surface reaction and diffusion, and multi-exponential models. However, both PFO and PSO have been the most common models applied over the last two decades in a wide variety of adsorption systems, such as in biomass systems, nanomaterials as adsorbent, in heavy metals and pharmaceuticals compounds adsorption [140]. These models gained major visibility in the study published by Ho and Mckay [141], who applied them in several adsorption datasets in their linear forms and concluded that PSO provides the best correlation of the experimental data, which has been corroborated along the past two decades [139,140]. Also, researchers [135,137,142] believe that the PFO model is more related to a physical adsorption process and PSO to a chemical adsorption process. However, other studies indicate that both models are related to the whole adsorption process, and that the kinetic modelling is strongly influenced by the adsorbent dosage and the initial concentration tested [20,117,143]. Most studies reported in Gao et al. [20], where several types of adsorbent physicochemical characteristics and hormone adsorption behaviour was summarized, concluded that the PSO model was the most fitted to explain the kinetic behaviour within the process. However, results on hormones adsorption kinetics using DWTR were not in total agreement with the PSO trend. According to Martins et al. [117], the fit of kinetic data was depended on the initial hormone concentration, and the data fit varied between the PFO, PSO and Elovich models. Even though, the equilibrium was reached between 3 and 8 h, being comparable to other studies related to the adsorption of hormones, such as those using activated magnetic biochars [144], bone charcoal [145], and biochars derived from agro-industrial waste [146].

3.3.2.3. Adsorption isotherm. The last step in modelling the adsorption process is to identify the main mechanisms responsible for the connection between adsorbate and adsorbent (in equilibrium conditions), using isotherm models. The mechanisms within this process include chemical adsorption (chemical bonds formation), physical adsorption (van der Waals forces) and ion exchange models [138]. These interactions can be identified using the most common isotherm models, such as Langmuir, Freundlich or Brunauer, Emmett, and Teller (BET) models, though there are several other models to describe adsorption mechanisms. The Freundlich model is an empirical isotherm used to describe nonlinear adsorption phenomena on heterogeneous surfaces and assumes a multilayer process. Due to its empirical nature, it lacks specific physical meaning [147,148]. On the other hand, Langmuir is a theoretical model, which settles on a chemical adsorption mechanism through a monolayer process where the adsorption sites are homogeneous [148,149]. Theoretically based physical adsorption models, like BET, represent a multilayer homogeneous process where the adsorption energy in the first layer differs from the subsequent layers and the adsorption rate is equal to the desorption rate. The BET model has also been applied for the determination of physical parameters such as pore size distribution and surface area [148,150]. The adsorption of the estrogen E2 was best fitted to the Freundlich model, whereas EE2 adsorption presented equal R^2 values for both Langmuir and Freundlich models [117].

Comparing DWTR to other types of adsorbent material, the Langmuir model best represented the adsorption of hormones onto biochar-based materials [120,135,137,145,151], graphene-based materials [136,152–154] and silica-based materials [156–159]. The Freundlich model provided the best fit for hormone adsorption using activated carbon [160,161], carbon nanotubes [134,162,163] and polymer-based materials [165,166], as summarized by Gao et al. [20]. The isotherm modelling of EE2 described by Martins et al. [117], where both Langmuir and Freundlich presented suitable fits, was also observed in the adsorption of E2 onto biochars derived from agricultural waste [146].

Due to its adjustment to both models, an interesting analysis was carried out by the authors, in which a parameter from Sips isotherm (hybrid model combining Langmuir and Freundlich) was considered, which predicts the heterogeneity of the surface to assess whether the process occurs mainly by Langmuir or Freundlich. Martins et al. [117] concluded that when the heterogeneity parameter is closer to one, the adsorption process occurs according to Langmuir isotherm, and for lower values, the Freundlich is more suitable.

3.4. DWTR adsorption mechanisms for different EPs

The reuse of DWTR as a low-cost adsorbent for phosphorous removal is undoubtedly a dense, well-documented topic, with many recent published investigations and reviews (Table 5). The strong affinity between phosphorous and Al-sludge is based on an inner-sphere reaction where phosphate replaces the functional group in the Al-sludge surface, meaning that the dominant adsorption mechanisms are by ligand exchange [49,87]. Therefore, there has been intense application of DWTR for phosphorous recovery from water to further application on soil remediation since it has become a scarce nutrient [167]. Although research efforts so far have focused on phosphorous removal, the potential application of DWTR as an adsorbent of pollutants from water environments is wider. The amorphous character of DWTR and the presence of surface functional groups have made this material compatible with metal ions, enhancing its potential for heavy and semi-metal ions removal (Table 6). The main mechanism for metal ions removal is by specific adsorption (bond between functional group and the heavy/semi-metal ion), non-specific adsorption (ion exchange and π-π interactions), surface precipitation (electron transference on the surface promoting the formation of more adsorption sites) and fixation (diffusion of metal ions through the porous structure). Such interaction between metal ions and DWTR make this material suitable for heavy metal immobilization in the environment [125]. Within the spectra of well documented compounds on the efficiency of DWTR as an adsorbent, dyes from the textile and similar industries are highlighted (Table 7).

Nonetheless, being aware of the current water treatment demands, regarding more recalcitrant compounds, the literature review regarding the use of DWTR (without modification) for the removal of EP, such as endocrine disrupters [94,117], antibiotics [130,203], pesticides [98,118,129,204] and surfactants [16] from water matrices are summarized in Table 8.

The adsorption behaviour varies for each compound, particularly in the case of hormones, which is the primary focus of this review. It has been observed that the adsorption process is influenced by interactions with activated sites, and these interactions are dependent on the concentration of adsorbates. Due to the hydrophobic nature of hormones, as previously discussed, this characteristic emerges as one of the predominant adsorption mechanisms. In the chemical structure of E1, E2, and EE2, there are components such as a benzene ring, a phenolic hydroxyl group, and a hydroxyl/ketone group. Consequently, the bonding

Table 5
Phosphorous adsorption using DWTR in water matrices.

Type of sludge	Pre-treatment	*qe* – Adsorption capacity (mg/g DWTR)	Solution pH	Reference
Al-DWTR	Pyrolyzed (700 °C, with N_2 supply to prevent oxidation)	33.75	6	
Al-DWTR	Pyrolyzed (500 °C, with N_2 supply to prevent oxidation)	34.53	6	[168]
Al-DWTR	Air-dried	30.83	6	
Al-DWTR	Oven-dried (105 °C)	15.06	6	
Al-DWTR	Air-dried	25	4	[127,169]
Al-DWTR	Air-dried	12.5	–	[127,170]
Al-DWTR	–	31.9	4	[127,171]
Al-DWTR	Dried at 50 °C for 5 days	15.57	4	[172]
Al-DWTR	Oven-dried at 105 °C for 24–48 h	32.27 to 42.67	6	[173]
Al-DWTR	Air-dried	20.1 and 22.4	4.3	[174]
Al-DWTR	Oven-dried at 103 °C	4.86	4	[175]
Al-DWTR	Dried for 2 h at 105 °C	25.33	5.5	[111]
PAC-DWTR	Dried for 48 h at 105 °C	1.25–1.50	–	[74]
Al-DWTR	Air-dried and oven at 105 °C for 24 h	0.37	4	[177]
		0.32	7	
Al-DWTR	Dried and sieved	2.57	7.4	[178]
Al-DWTR	Oven-dried at 105 °C for 48 h and sieved to 1.18 mm	0.35	6	[179]
Al-DWTR	Air-dried	36.6	7	[180]
		34.1	7	
		27.6	7	
		19.5	7	
Al-DWTR	Sieved into 1–4 mm and dried at 550 °C for 2 h	7.27	7	[181]
PAC-DWTR	150	0.120	–	[96]
PAC-DWTR	No modification	0.115	–	
PAC-DWTR	No modification	1.27	5.9	[115]
Fe-DWTR	Air-dried for 3 weeks and then grounded and sieved into a diameter < 2 mm.	4.76	3.9	[182]
Al-DWTR	Sieved into 1 mm mesh, then oven dried at 105 °C for 24 h.	3.673	6.96	[183]

Table 6
Heavy metal adsorption using DWTR in water matrices.

Pollutants	Type of sludge	Pre-treatment	*qe* adsorption capacity (mg/g DWTR)	Solution pH	Reference
As (III)	Fe-DWTR (with manganese content)	Air-dried	36.53	7	[184]
		Dried for 72 h at 25 °C	132.17	7.7	[99]
	Fe-DWTR	Air-dried	19.43	6	[186]
	Al-DWTR	Air-dried	69.77	6	
As (V)	Fe-DWTR (with manganese content)	Air-dried	40.37	7	[184]
	Fe-DWTR (with manganese content)	Dried for 72 h at 25 °C	76.73	7.7	[99]
	Fe-DWTR (with manganese content)	Oven-dried for 24 h at 105 °C	42.9	8.1	[187]
	Al-DWTR	Air-dried	3.3, 5.0 and 50 (depending on the granulometry-the lowest has the higher *qe*)	7.2	[107]
	Fe-DWTR	Air-dried	22.79	6	[186]
	Al-DWTR	Air-dried	124.02	6	
	Fe-DWTR	Air-dried	40.98	6.5	[188]
Cr	Fe-DWTR	Dried for 2 h at 550 °C	89.12	3	[189]
Cr (III)	Al-DWTR	Air-dried	88.1–121.4	5	[190]
Cr (VI)	Al-DWTR	Air-dried	34.01–35.63	5	[190]
		Dried at 105 °C for 2 days	1.62	6.38	[191]
Co	Al/Fe-DWTR	Air-dried	17.31	6	[192]
Cu	Al-DWTR	Dried at 100 °C for 24 h	35–40	6.6	[193]
	Al-DWTR	Air-dried	10	–	[194]
	Al-DWTR	Dried at 105 °C for 12 h	3.494	4.5	[125,195]
	Fe-DWTR	Dried at 105 °C for 12 h	3.496	4.5	[125,195]
	Al-DWTR	Air-dried	93.9	6	[196]
Pb (II)	Al-DWTR	Dried for 2 h at 105 °C	8.05	5.5	[111]
	Al-DWTR	Dried at 105 °C for 12 h	12.873	4.5	[125,195]
	Fe-DWTR	Dried at 105 °C for 12 h	12.873	4.5	[125,195]
	Fe-DWTR	Dried for 2 h at 550 °C	9.93	3	[189]
	Al-DWTR	Air-dried	141.8	6	[196]
	Al-DWTR	Air-dried	86.1–99.4	5	[190]
Ni	Fe-DWTR	Dried for 24 h at 105 °C	6.97	5	[197]
	Fe-DWTR (with manganese content)	Dried for 24 h at 105 °C	11.6	6.5	[198]

interactions between hormones and the active sites in the adsorbent material are primarily based on phenyl or hydrogen surface groups, thereby enhancing hydrophobicity [20]. Alongside with this mechanism, both π-π interaction, hydrogen bounding and electrostatic interactions play an important role in the hormone adsorption process, as discussed in the previous section.

The tetracycline adsorption process is controlled by intra-particle diffusion and surface adsorption, in which the dominant mechanisms

Table 7
Dyes adsorption using DWTR in water matrices.

Pollutants	Type of sludge	Pre-treatment	*qe* – Adsorption capacity (mg/g DWTR)	Solution pH	Reference
Congo Red	PAC-DWTR	The sample was subjected to oven drying at 105 °C for 24 h, followed by grinding using a mortar and subsequent sifting through a 60-mesh sieve	21,00	Not adjusted	[130]
		The sample was dried in an oven at 105 °C for 24 h, then ground using a mortar and sifted through a 60-mesh sieve. Subsequently, it was subjected to pyrolysis at 400 °C for 4 h.	44.504	7	
Indigo Carmine (IC)	Al-DWTR	The sample was air-dried and sieved through 2 mm mesh.	38.86	5	[106]
	Al-DWTR[a]	Air-dried and sieved through 51 μm mesh.	172.4	5	
Sanodure Green (SG)	Fe-DWTR	Stored at room temperature (20–25 °C), grounded into mortar	62.93	2	[199]
Disperse Blue 79	Al-DWTR	Sun-dried	1.315	3	[200]
Basic Violet 16	Fe-DWTR	Slurry form	3333.34	5	[201]
Acid Blue 40	Fe-DWTR	Slurry form	833.34	5	
Direct Blue 71	Fe-DWTR	Slurry form	625	5	
Methylene blue	PAC-DWTR	Dried for 48 h at 105 °C	1–1.25	–	[74]
Acid red 97	Al-DWTR	Dried for 8 h at 120 ° C, then dried at 600 °C for 2 h and sieved to <0.147 mm	2238.6503	3	[202]

[a] DWTR nanoparticles.

are complex precipitation and non-specific adsorption (ion exchange, π-π interaction and hydrogen bonding). The ion exchange dependence made this process ionic strength dependent, being observed in conditions with higher initial concentrations. Also, there may be a possibility of forming a mononuclear monodentate surface complex through a strong inner-sphere bond [130,205]. Pesticides such as chlorpyrifos showed to have a great affinity for DWTR, being the adsorption process mainly controlled by hydrogen bonding, electrostatic interaction between Fe and Al ions and finally through the formation of complexes with these species [129]. Likewise, in the case of chlorpyrifos, the adsorption process of thiamethoxam indicates that the underlying mechanisms involve hydrogen bonding, the formation of complexes with aluminium and iron, and electrostatic interactions [118]. Surfactants such as perfluorooctanoic acid (PFOA) and perfluorooctanesulfonic acid (PFOS), also showed a high affinity for Al-DWTR, as they immobilize both compounds through the formation of inner or outer-sphere complexes surface functional groups and/or hydrophobic interactions. Specifically, the formation of inner-sphere complexes through ion exchange can result in robust adsorption with minimal desorption, ensuring a safe process with no risk of leaching [16].

3.5. Limitations of DWTR application

The adsorption capacity (qe) parameter establishes a relation between the adsorbate concentration and the adsorbent dosage under equilibrium conditions. However, due to the temporal-spatial variability in the physicochemical properties of DWTR, as mentioned above, the adsorption capacity is also variable and difficult to predict, which may explain the variability in its reported values. This variability is a limitation of the process itself due to the heterogeneous nature of DWTR. In addition, the dependence on the external conditions of the adsorption reactions, such as the solution pH, temperature, competition between adsorption sites, adsorbent dosage and initial adsorbate concentrations, can affect the adsorbent capacity [47,127,206].

Other limitations related to the use of DWTR include the leaching of metal ions and other organic micropollutants that were adsorbed during the water treatment process [125,207,208]. The most common methods used in the literature to assess the leaching character of materials are the toxicity characteristic leaching procedure (TCLP, US EPA Method 1311) and the leaching environmental assessment framework (LEAF, US EPA Method 1313, 1314, 1315 and 1316) [53,125]. Additionally, ecotoxicological tests are useful to identify potential risks to the receiving environment [209,210]. Metals in DWTR are normally stable at neutral and alkaline conditions and leachable at acidic conditions [125]. The common presence of aluminium (Al) in DWTR also raises concern due to its potential leaching risk, as aluminium is a neurotoxic compound responsible for brain and bone structure disorders [211]. A recent study [115] on the application of Al-DWTR and PAC-DWTR indeed showed Al leaching. However, the TCLP applied by these authors showed that these leaching values led to Al concentrations within the acceptable range (0.63 to 3200 μg/L) according to the US quality criteria for the presence of Al in freshwater under the Clean Water Act [212]. Lee et al. [74] also applied the TCLP to access the stability of a PAC-DWTR during a pyrolysis process from 200 to 700 °C. The amount of leached aluminium increased with increasing temperature, being more expressive above 400 °C. However, after this physical regeneration process, PAC-DWTR, when tested in water, increased its phosphate and methylene blue adsorption capacity compared to the raw material without further leaching. Therefore, both studies referred to the suitability of this material for reuse and multi-functional application [74,115]. Supporting the latter, a recent study on DWTR in the context of circular economy (CE) developed by Nguyen et al. [51], where three main benefits are pointed out that can be derived from DWTR reuse. The first is the environmental benefit, well explored in this review, by mentioning the several alternatives of application in Section 3.2. The second advantage of DWTR in the CE management approach is of economic nature, by reducing the cost of raw materials if DWTR can be effectively reused and replace the virgin material (for the construction sector or as a coagulant). However, the main economic benefit arises from environmental legislation and taxes involved in the disposal of this material. Finally, the social benefit, where the implementation of CE approaches can lead water authorities the recognition as innovators in sustainability, creating a sustainable business image, which drives into a major sense of community and cooperation. Also, the CE management approach will provide a closer link between water entities' objectives and sustainable development goals achievement [51].

Table 8
DWTR used for micropollutants adsorption in water matrices.

Pollutants	Type of sludge	Detection method	Pre-treatment	q_e – Adsorption capacity (μg/g DWTR) + C_0 initial concentration	Solution pH	Location	Reference
17β-Estradiol (E2)	Al-DWTR	High-performance liquid chromatography coupled with a fluorescence detector (HPLC-Fl)	Dried at 100 °C, sieved (0.35 mm) and carbonized at 550 °C	0.035 ± 0.001 (0.5 μg E2/L) 5.830 ± 0.216 (100 μg E2/g)	5.5	Brazil	[117]
	PAC-DWTR	High-performance liquid chromatography tandem mass spectrometry (HPLC-MS-MS)	Air dried	0.108 (0.5 μg E2/L) 0.064 (0.35 μg E2/L) 0.05 (0.2 μg E2/L)	3	Portugal	[94]
	PAC-DWTR		Air dried	0.11 (0.5 μg E2/L) 0.07 (0.35 μg E2/L) 0.05 (0.2 μg E2/L)			
17α-Ethinylestradiol (EE2)	Al-DWTR	High-performance liquid chromatography coupled with a fluorescence detector (HPLC-Fl)	Dried at 100 °C, sieved (0,35 mm) and carbonized at 550 °C	0.043 ± 0.001 (0.5 μg EE2/L) 6.477 ± 0.593 (100 μg E2/g)	5.5	Brazil	[117]
	PAC-DWTR	High-performance liquid chromatography tandem mass spectrometry (HPLC-MS-MS)	Air dried	0.09 (0.5 μg E2/L) 0.026 (0.35 μg E2/L) 0.04 (0.2 μg E2/L)	3	Portugal	[94]
	PAC-DWTR		Air dried	0.11 (0.5 μg E2/L) 0.05 (0.35 μg E2/L) 0.05 (0.2 μg E2/L)			
Tetracycline (TTC) and oxytetracycline (OTC)	Al-DWTR	High-performance liquid chromatography coupled with photodiode array (HPLC – PDA)	Air-dried and sieved to 2 mm	300.3 (5 mM of TTC and OTC)	6	United States of America - Florida	[205]
Tetracycline (TTC)	PAC-DWTR	UV–Vis spectrophotometer	Oven-dried at 105 °C for 24 h and ground with a mortar and sifted through a 60-mesh sieve.	15,000 (100 mg TTC/L)	Not adjusted	China	[130]
			Oven-dried at 105 °C for 24 h and ground with a mortar and sifted through a 60-mesh sieve and pyrolyzed at 400 °C for 4 h	45,455 (180 mg TTC/L)	4		
Chlorpyrifos (CPF)	Al-DWTR[a]	UV–Vis spectrophotometer (wavelength 289 nm) and High-performance liquid chromatography coupled with UV detector for confirmation.	Air-dried and sieved with pores of 2 mm and 51 μm, then mechanically ground to <100 nm	4500 (25 μg CPF/L)	7	Egypt	[129]
	Al-DWTR	High-performance liquid chromatography coupled with UV detector (wavelength of 288 nm).	Air-dried, gently crumbled and sieved through a 0.15 mm mesh	465 (210 μg CPF/L)	7.07	China	[98]
Glyphosate	Al-DWTR	Determined through total phosphorus measurement, using a theoretic linear relationship between total phosphorus and glyphosate. Total phosphorous determination by UV–Vis spectrophotometer	Air-dried and grounded into <0.063 mm	9600 (50 mg glyphosate/L)	5.6	Ireland	[204]
Thiamethoxam (TMX)	Al-DWTR	UV–Vis Spectrophotometer (range 190–400 nm)	Air-dried and sieved to 2 mm mesh	0.019 (3.5 mg TMX/L)	7	Egypt	[118]
	Al-DWTR[a]		Air-dried and sieved to <100 nm mesh	0.05 (3.5 mg TMX/L)			
Perfluorooctanoic acid (PFOA)	Al-DWTR	High-performance liquid chromatography tandem mass spectrometry (HPLC-MS-MS)	Air dried and sieved to 850-μm and then milled to micron-sized powders	97 (1.0 mg PFOA/L)	3	United States of America – New Jersey	[16]
Perfluorooctanesulfonic acid (PFOS)	Al-DWTR			100 (1.0 mg PFOS/L)			

[a] DWTR nanoparticles.

4. Conclusions

This review aimed to provide a new perspective on the use of DWTR as an adsorbent material for hormone removal. Management of water sector by-products remains a major challenge, although the possibility of fully reusing the main residual as a value-added material or even as a new product is a step forward towards a more sustainable approach and the application of the circular economy principles in the water sector. Overall, some final remarks must be addressed. DWTR are extensively reported to be effective in the adsorption of phosphate and heavy metals, however, there is a lack of knowledge on a wider application of DWTR, such as their adsorption of hormones.

This fact presents a limitation within this review, as only one comprehensive study has been conducted. However, it also represents a potential opportunity for further research in this field.

Although the studies carried out so far indicate that DWTR has a significant adsorption capacity for various types of EPs, this presents an optimistic outlook for the potential reuse of the material as an adsorbent. Looking ahead to prospective applications, it is crucial to prioritize further research into EP adsorption within wastewater matrices,

particularly as WWTPs represent the primary source of these compounds into the environment. Given the variable composition of wastewater, this presents a challenge to address.

Moreover, since adsorption is a phase transition process rather than complete elimination, it is imperative to consider the fate of DWTR after their initial use, given that they could be saturated with hazardous compounds. As such, carrying out a cost estimation analysis becomes essential to determine whether a reactivation or final disposal process is more viable from an economic and environmental point of view.

There are a few studies on the characterization of PAC-DWTR. Considering the decreasing quality of water bodies and the need for PAC application on raw water treatment, such type of DWTR should be further explored due to the possibility of AC adsorption availability after water treatment. DWTR heterogeneity and how this may affect the variability of results should also be evaluated in future works. Finally, further studies on the application of DWTR for the removal of EP, such as hormones, should be supported not only by standard toxic leaching procedures but also by ecotoxicological assessments.

Supplementary data to this article can be found online at https://doi.org/10.1016/j.jwpe.2023.104322.

CRediT authorship contribution statement

Rita Dias: Conceptualization, Original draft preparation. **Michiel A. Daam**: Writing - Review & editing. **Mário Diniz**: Writing - Review & editing **Rita Maurício**: Writing - Review & editing.

Declaration of competing interest

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

Data availability

No data was used for the research described in the article.

Acknowledgements

The authors acknowledge and thank the support given to CENSE by the Portuguese Foundation for Science and Technology (FCT) through the strategic project UIDB/04085/2020 and through a PhD grant by FCT for Rita Dias (SFRH/BD/148793/2019). The projects UIDP/04378/2020 and UIDB/04378/2020 of the Research Unit on Applied Molecular Biosciences - UCIBIO and the project LA/P/0140/2020 of the Associate Laboratory Institute for Health and Bioeconomy - i4HB are also acknowledged.

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