



Key Properties of Drinking Water Treatment Residuals for their Effective Reuse as Phosphate Adsorbents

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Abstract

Drinking Water Treatment Residuals (DWTR) are the sludge by-products produced in the drinking water treatment plants, representing a significant amount of the operational costs. Since the DWTR properties depend on the treatment system and the raw water characteristics, it is crucial to understand the key parameters to streamline the decision-making process regarding DWTR reuse. This study aimed to assess the key parameters of DWTR as an adsorbent material of phosphate (PO_4^{3-}).

To this end, seven different DWTR were subjected to physical and chemical characterization. Adsorption tests were carried out under neutral pH. Following a comprehensive analysis of the correlation between the quantitative properties and the adsorption capacity, a reliable predictor has been determined. This predictor will inform the construction of a model that will accurately predict the adsorption capacity.

The characterization showed differences among the DWTR, supporting the influence of the DWTR properties. Six of the DWTR were able to adsorb PO_4^{3-} , with adsorption capacities ranging between 6.7 and 44.1 mg/g. Contrary to expectations, the aluminium content was not the key parameter for the adsorption process. Instead, the crucial factor was the pH at the point of zero charge (pH_{PZC}). Based on this relationship, a statistical model was built, using pH_{PZC} as a predictor of adsorption capacity (p -value < 0.0001, $R^2 = 0.735$).

The use of pH_{PZC} as a predictor of the PO_4^{3-} adsorption capacity of DWTR enables the reduction of the number and the costs of analytical determinations and streamlines the decision-making process for stakeholders regarding DWTR reuse.

Highlights

- Seven Drinking Water Treatment Residuals (DWTR) were fully characterized.
- Phosphate removal is not dependent on aluminium content.
- The pH at the point of zero charge (pH_{PZC}) is a key parameter in phosphate adsorption.
- Water utilities can use pH_{PZC} to assess the adsorption capacity of DWTR.
- The model predicts 73.5% of phosphate adsorption capacity onto DWTR.

Keywords Drinking water treatment sludge reuse · Sludge characterization · Phosphate adsorption · Water treatment

Extended author information available on the last page of the article

1 Introduction

Drinking water treatment involves processes such as coagulation and flocculation that inevitably produce solid waste, commonly referred to as sludges. These sludges are also known as Drinking Water Treatment Residuals (DWTR) (Zhao et al. 2021). Drinking Water Treatment Plants (DWTP) globally produce 10 000 t/day of DWTR (Carleton and Cutright 2020). DWTR are typically composed by minerals, organic matter, microorganisms, and chemicals reagents used in the drinking water treatment (e.g. hydrated metal oxides of Al and Fe) (Pająk 2023; Dias et al. 2023). Based on the reagents used in the water treatment process, DWTR can be classified as aluminium-rich (Al-DWTR) or iron-rich based (Fe-DWTR) (Kulandaivelu et al. 2020). However, the global decline trend in surface water quality has prompted the increased use of powdered activated carbon (Bonilla-Petriciolet et al. 2019), leading to a distinct sludge type known as activated carbon-rich DWTR (AC-DWTR).

The production of DWTR is expected to rise since population growth and urban expansion will drive higher demand for drinking water. Therefore, adopting a sustainable approach to DWTR management is essential, as its disposal entails the loss of a by-product with potentially valuable applications across various sectors (Nguyen et al. 2022b). Moreover, DWTR contributes significantly to DWTP's operational costs (Shen et al. 2019; Nayeri and Mousavi 2022). For instance, in Victoria, Australia, the disposal of DWTR incurs an annual cost of approximately \$6.2 million (Nguyen et al. 2022b). Furthermore, increasingly stringent environmental regulations aim to reduce landfill disposal, reinforcing the need to reconsider current DWTR management practices (Nayeri and Mousavi 2022; Nguyen et al. 2022b).

In this context, DWTR is progressively being redefined—not as waste, but as a valuable by-product—an evolution supported by the principles of the circular economy in the water sector (Lee et al. 2020; Rahmati et al. 2022; Nguyen et al. 2022b). This by-product can be used for different purposes such as aggregates in the construction sector, soil improvement agents, environmental remediation, used as a coagulant due to the high content of coagulants, and as substrate in constructed wetlands (Shen et al. 2019; Dias et al. 2023). Among its various potential applications, DWTR has been identified as a promising adsorbent material, owing to its chemical composition and porous structure (Pająk 2023; Dias et al. 2023). Therefore, adsorption studies have been conducted to assess the DWTR adsorption capacity for several chemical species such as heavy metals (Arab et al. 2022; Pająk 2023), dyes (Gadekar and Ahammed 2020), compounds of emerging concern (Kulandaivelu et al. 2020; Dias et al. 2021) and phosphorus (Zhao and Yang 2010; Bal Krishna et al. 2016).

Specifically, the need for phosphorus adsorption arises from its common presence in water bodies, where it contributes to eutrophication, a process that poses significant risks to aquatic ecosystems (Muisa et al. 2020). Several studies have already assessed the adsorption of phosphorus (P) using DWTR, reaching positive results on the process (Muisa et al. 2020; Carleton et al. 2020). For example, Zhao and Yang (2010) reported that phosphorus adsorption increases with the pH decrease. In the study of Lee et al. (2020), it was reported that AC-DWTR can be used for the removal of anions and cations simultaneously. Hou et al. (2018) reported that the phosphorus adsorption onto the DWTR was due to the bound to Calcium, Aluminium and Ferrous present in the DWTR. Van Truong and Kim (2021) studied the use of pyrolyzed Al-DWTR, for the removal of phosphate. Their research demonstrated that the adsorption process was primarily due to chemisorption, as indicated by

the results of conventional isotherm models. Everaert et al. (2021) studied various calcination treatments for the reuse of DWTR for phosphorus. Their study found that the highest adsorption capacity, measured at 7.3 mg/g, was achieved with a pre-treatment at 550 °C. According to the research conducted by Nguyen et al. (2022a), phosphate adsorption onto DWTR aligns with the Langmuir isotherm model. The study indicates that aluminium content plays a pivotal role in the adsorption process. However, some proprieties such the pH at the point of zero charge were not assessed.

Although few studies have investigated which specific properties of DWTR are responsible for its adsorption capacity. Such conclusion was referred by Rahmati et al. (2022), highlighting the importance of understanding the properties of DWTR that make them suitable for phosphorus adsorption. The reuse of DWTR is not only an environmentally friendly solution but also offers economic benefits. As mentioned above, the reuse of DWTR reduces their disposal costs, but also the costs associated with purchasing raw materials for phosphorus removal (Nguyen et al. 2022b). For instance, phosphorus removal costs can range between 37 and 118 \$/kg of P removed (Bashar et al. 2018). Additionally, this potential for reuse would reduce the costs associated with landfilling (Siswoyo et al. 2014; Nguyen et al. 2022b). For example, in the study made by Bensitel et al. (2023) the reuse of DWTR as a low-cost coagulant in one DWTP resulted in a reduction of the amount of coagulant required per year to one-third.

DWTR presents an opportunity for applying the circular economy approach, particularly in phosphorus removal from water. To promote the reuse of this by-product, it is crucial to understand the key properties that influence phosphate adsorption capacity (Bensitel et al. 2023). As mentioned above, the ability of DWTR to adsorb phosphate has been studied, but most studies focus on the use of conventional adsorption models, reporting only the maximum adsorption capacity. Although such studies are important, there is a need to look for the key properties of the sludge, in a pragmatic way, that may indicate easily its suitability for use as a phosphate adsorbent. Furthermore, the DWTR complete characterisation can be expensive and requires specialised equipment and technicians. Therefore, simplifying the selection of DWTR is crucial to promote its reuse (Rahmati et al. 2022).

Nevertheless, it should be noted that certain parameters that influence adsorption processes related to surface chemistry and textural properties were not monitored in this study. Based on this knowledge gap this study aims to identify the key properties of AC-DWTR that can reliably and easily predict its phosphate adsorption capacity. This knowledge will enable a streamlined and cost-effective selection process for water and wastewater treatment applications. Furthermore, it empowers water and wastewater utilities with the necessary methods and knowledge to manage this by-product and explore its potential integration into advanced wastewater treatment systems. To this end, seven AC-DWTR from different DWTPs were subjected to a characterization of their physical and chemical properties. These properties were correlated with the adsorption experiment to identify which properties can be used to predict whether the DWTR is suitable for removing phosphorus in other to streamline the decision-making process.

2 Materials and Methods

2.1 Drinking Water Treatment Residuals

Seven AC-DWTR were collected from different DWTPs after the final stage of dewatering. The selection of the WTP was carried out based on the characteristics of the raw water, the river basins in which they are located, and the drinking water treatment systems applied. These criteria allowed the selection of DWTR with different properties. Table SM1 in the supplementary material shows the DWTPs specification for each DWTR. All the seven DWTR have in common the reagents used in the drinking water treatment, namely polyaluminium chloride, milk of lime, activated carbon (AC), and polyelectrolyte. The sampling campaign was conducted over one day at each DWTP. The samples were collected in boxes at the outlet of the dewatering stage in. After collection, the samples were left at room temperature for one week and then dried at 105 °C to remove all residual water. The dried material was ground and sieved to obtain a particle size of 45/60 mesh (250–354 µm) and the sieved samples were stored in a desiccator.

2.2 Drinking Water Treatment Residuals Characterisation

The elemental analysis was performed to quantify the carbon, hydrogen, nitrogen, and sulphur content using an Elemental Thermo Finnigan Analyzer—CE Instruments, model Flash EA 1112 CHNS series (Waltham, MA, USA), based on sample combustion dynamics. The ash content of the DWTR was determined according to the ASTM D1762-84 (2013) guideline (750 °C). The pH_{PZC} was carried out following the method described by Bernardo et al. (2020): this method is based on the variation of the initial pH, and the determination of the pH_{PZC} was defined as the point on the curve ΔpH ($\Delta\text{pH} = \text{final pH} - \text{initial pH}$) versus initial pH, where ΔpH is equal to zero. The thermogravimetric analysis (TGA) was conducted in Setaram Labsys EVO equipment (Caluire, France) between room temperature and 900 °C with a heating rate of 5 °C/min under argon atmosphere. The mineral content was performed by inductively coupled plasma atomic emission spectroscopy (ICP-AES) (Horiba Jobin-Yvon equipment, Kyoto, Japan) after acidic digestion. The acidic digestion was performed using aqua regia (1 HNO_3 :3 HCl v/v) at a microwave ETHOS 1 - Advanced Microwave Digestion System (Milestone, Sorisole, Italy) (EN 13346 2000). The mineral analysis was carried out for the quantification of the following elements: Al, Ca, Cr, Cu, Fe, K, Mg, Na, P, Pb and Zn. Fourier Transform Infrared Spectroscopy (FTIR) analysis was conducted using the KBr disk method with a Perkin-Elmer – Spectrum 1000 Spectrometer (Massachusetts, USA) in the range between 4 000 to 400 cm^{-1} under a resolution of 1 cm^{-1} . The textural analysis was performed through N_2 adsorption experiment at 77 K (ASAP 2010 Micromeritics equipment, Atlanta, GA, USA). The BET surface area (S_{BET}) was calculated following the Brunauer-Emmett-Teller model with the modifications proposed by Rouquerol et al. (2007). The total pore volume (V_{total}) was determined at a relative pressure of $p/p_0 = 0.95$. The microporosity volume ($V_{\text{micropores}}$) was estimated by applying the t-plot method. The volume of mesopores ($V_{\text{mesopores}}$) was determined from the difference between the V_{total} and $V_{\text{micropores}}$. The pore size distribution (PSD) was analyzed by using the Non-Local Density Functional Theory (NLDFIT). The morphology and elemental composition of the DWTR surface were analysed by Scanning Electron Microscopy with Energy Dispersive Spectros-

copy (SEM-EDS), using a JEOL 7001 F analytical FEG-SEM with Oxford model INCA 250 PREMIUM EBSD (electron backscatter diffraction) and energy dispersive X-ray spectrometer light elements detector attachments.

The leaching assay was performed according to the European standard leaching test for waste materials and sludges (EN 12457-2:2003 (2003)). The samples with a liquid/solid ratio of DWTR of 10 L/kg were submitted to agitation in glass bottles at 10 rpm in a rotax 6.8 overhead shaker (VELP Scientifica Srl, Usmate, Italy) for 24 h. The leachates were filtrated through cellulose nitrate membranes of 0.45 μm membrane filter (MF-Millipore, Merck, Darmstadt, Germany) under vacuum and the obtained eluates were characterized for pH (inoLab pH/ION 735, Weilheim, Germany), conductivity (inoLab Cond7110, Weilheim, Germany), Cl^- and SO_4^{2-} . The Cl^- and SO_4^{2-} content was according to the method established for the column AS11-HC 4-mm and AG11-HC 4-mm Analytical & Guard Columns in an Ion Chromatograph DIONEX ICS-3000 (Sunnyvale, Ca, USA). The eluates were also characterized for metallic species to assess their aqueous mobility. To this end, a fraction of the eluates was acidified with HNO_3 (1 N) to $\text{pH} < 2$ and the content was quantified by ICP-AES. The relative mobility of the chemical elements was determined by Eq. (1).

$$\text{Relative mobility (\%)} = \frac{\text{Concentration in aqueous eluates}}{\text{Concentration in DWTR}} \times 100 \quad (1)$$

2.3 Phosphate Adsorption Experiment

The stock solution of phosphate was prepared with a concentration of 400 ppm using potassium dihydrogen phosphate (KH_2PO_4) (Merck, Darmstadt, Germany) in distilled water, from which working solutions with 50 mg/L of PO_4^{3-} were prepared in distilled water. For the adsorption experiments, 0.1 g of DWTR (particle size of 45/60 mesh) was added to 200 mL of the working solution. The solutions with DWTR were submitted to agitation (230 rpm) in a shaker (HS 501 digital, IKA-Werke, Staufen, Germany) for 24 h. After mixing, the samples were filtered through 0.45 μg membrane filter (MF-Millipore, Merck, Darmstadt, Germany) under vacuum. The remaining PO_4^{3-} present in the filtered samples was determined according to the ascorbic method (4500-P E.) described in American Public Health Association (1998), using a spectrophotometer Merck Spectroquant Prove 100 (Merck, Darmstadt, Germany). Based on this method, a calibration curve was obtained Eq. (2). This was applied to the $\lambda = 880 \text{ nm}$ ($R^2 = 0.997$; Predicted $R^2 = 0.997$; Limit of Determination (LOD) = 0.07 mg/L; Limit of Quantification (LOQ) = 0.22 mg/L).

$$\text{Absorbance} = 0.0121 \times \text{PO}_4^{3-} + 0.0068 \quad (2)$$

The amount of PO_4^{3-} adsorbed was calculated using Eq. (3), where q_e is the amount of adsorbate adsorbed (mg/g), C_0 is the initial adsorbate concentration (mg/L), C_e is the final adsorbate concentration (mg/L), V is the sample volume (L) and m is the mass of DWTR (g).

$$q_e = \frac{(C_0 - C_e)V}{m} \quad (3)$$

2.4 Modelling: Selection of Predictor and Model Validation

The selection of the predictor of the DWTR adsorption capacity was performed through a correlation between the q_e value (obtained in the adsorption experiment) and the following DWTR properties: pH_{PZC} , S_{BET} , Al, C, O, N, Ca, P and Fe content. The selection of these properties was based on the fact that these properties are the most frequently identified in the literature as having an influence on the adsorption process. The parameter with the highest correlation coefficient was identified as the primary predictive variable. A subsequent linear model was formulated based on this predictor, to determine the adsorption capacity of DWTR. The data analysis was performed using R software.

The developed model was validated by studying two sludges previously characterized in Rita et al. (2021)'s study. To this end, the sludge from this research was subjected to an adsorption test under the same conditions described in Sect. 2.3. The experimental data from this experiment was compared to the predictions made by the model.

3 Results and Discussion

3.1 Drinking Water Treatment Residuals Characterisation

The ash content, elemental analysis and the pH_{PZC} of the seven DWTR are shown in Table 1. The ash content of the DWTR samples ranged between 47.6% (DWTR6) and 62.1% (DWTR1). The differences between the DWTR may be explained by the amount of reagents used in the drinking water treatment (Dias et al. 2021). The ash content obtained was similar to the ones reported in the literature for AC-DTWR (Lee et al. 2020; Dias et al. 2021), but lower when compared to the ash content of the Al-DWTR (higher than 80%) used in Hou et al. (2018) study. The carbon content results revealed differences within the DWTR. According to Dahhou et al. (2023), the typical DWTR carbon content ranges between 14 and 18%, however, Siswoyo et al. (2014) found that the carbon content can be lower than 9%. Four of the studied DWTR had a carbon content higher than 18%, namely DWTR 2, 5, 6 and 7, this may suggest that these four DWTR have higher AC content incorporated (Dias et al. 2021). However, it is important to note that this does not necessarily imply that DWTR with higher carbon content have a higher amount of AC incorporated since the carbon content can be associated with both AC content and organic matter content (Lee et al. 2020).

Table 1 Ash content, elemental analysis, pH at the point of zero charge (adapted from Sousa et al. (2024))

Parameter	DWTR1	DWTR2	DWTR3	DWTR4	DWTR5	DWTR6	DWTR7
Ash (w/w%) ^a	62.1±0.3	58.4±0.4	58.2±0.1	56.2±0.2	50.3±0.7	47.6±0.5	48.5±0.7
C (w/w%)	7.03	21.67	16.37	13.86	20.57	18.99	20.73
H (w/w%)	2.10	2.55	1.66	1.32	2.99	3.15	2.05
N (w/w%)	0.17	0.74	0.32	0.37	0.75	0.96	0.71
S (w/w%)	0.00	0.00	0.00	0.00	0.00	0.00	0.00
O (w/w%) ^b	28.6	16.6	23.5	28.3	25.4	29.3	28.0
pH_{PZC}	11.3	5.5	9.2	8.4	6.5	3.8	8.4

^aMean ±SD, $n=2$

^bCalculated by difference: $100 - (C+H+N+S+Ash)$

The pH_{PZC} plays a crucial role in adsorption processes since it can either enable or inhibit the interactions between the adsorbent and the adsorbate, driven by electrostatic forces and complexation reactions (Pellenz et al. 2023). The DWTR 1 and 3 had an alkaline pH_{PZC} , which may be related to its high ash content and, consequently, higher mineral content (Dias et al. 2021). In contrast, DWTR2 and 6 exhibited acidic properties with pH_{PZC} values of 5.5 and 3.8, respectively, indicating a higher concentration of acidic surface groups in these DWTR (Bernardo et al. 2020). This may be related to the incorporation of organic acids in its composition derived from the presence of organic matter in the raw water. The remaining four DWTR exhibited neutral to slightly basic pH_{PZC} , consistent with the typical pH_{PZC} of DWTR (Castaldi et al. 2014; Muisa et al. 2020).

The textural analysis results (see Table 2) showed that the DTWR's surface area (S_{BET}) ranged between 16.1 and 217 cm^2/g , these results were similar to those found in several studies using this material (Shen et al. 2019; Lee et al. 2020; Dias et al. 2021; Jo et al. 2021). The ash and the carbon content often can be indicators of an adsorbent's surface area. Lower ash content is usually associated with higher surface area (Bernardo et al. 2020), while higher carbon content is potentially linked to higher surface area (Siswoyo et al. 2014). In the majority of the DWTR studied, this correlation can be identified (see Table 1). However, it is noteworthy that DTWR6 had the lowest surface area (16.1 cm^2/g), despite its low ash content and relatively high carbon content. The total pore volume of DWTR6 was also the lowest when compared to the other studied DWTR. Since the blocking of the porosity results in low surface area and volume pore (Salvador et al. 2015), the DWTR6's textural results may suggest a higher pore blockage. The PSD results (Figure SM1) also support this possibility. DWTR1 and 6 (Figure SM1 a) and f) respectively) had low pore volume within the micropore regions and higher volume for mesopores (>2 nm), while the other DWTR had a higher volume of micropores (<2 nm), and particularly samples 2, 3, 5, and 7 presented narrow micropores (<1 nm).

The results of the mineral analysis (Table SM2 of the Supplementary Material) revealed discernible differences among the DWTR, likely influenced by variations in raw water composition and the choice of drinking water treatment reagents. However, it was possible to identify that the elements present in higher concentration were calcium (Ca), magnesium (Mg) and aluminium (Al), as expected (Bal Krishna et al. 2016; Hou et al. 2018; Dias et al. 2021). The majority of the values obtained for the Al content are within the typical range for DWTR (12.8 to 174.6 mg/g) (Pajak 2023). However, DWTR 5 and 6 exceed this range, with values of approximately 188 mg/g . The high content of Al is likely related to the type of coagulant used in the drinking water treatment, namely, polyaluminium chloride. While the Ca and Mg content may reflect the characteristics of the raw water and the treatment applied to adjust water hardness. Additionally, the Ca content could also be explained by the incorporation of insoluble limestone residues into the DWTR (Dias et al. 2021).

Table 2 Textural properties (surface area and pores volume) of the DWTR

Parameter	DWTR1	DWTR2 ¹	DWTR3	DWTR4	DWTR5	DWTR6	DWTR7
S_{BET} (m^2/g) *	19.6	165	96.6	58.2	217	16.1	112
V_{total} (cm^3/g) *	0.044	0.154	0.137	0.085	0.243	0.009	0.067
$V_{\text{mesopores}}$ (cm^3/g)	0.043	0.114	0.120	0.077	0.194	0.004	0.028
$V_{\text{micropores}}$ (cm^3/g)	0.001	0.040	0.017	0.008	0.049	0.005	0.039

* Adapted from Sousa et al. (2024)

¹ Adapted from Sousa et al. (2025)

The TGA results (see Fig. 1) showed the DWTR thermal decomposition. These results offer insight into the behaviour of DWTR during activation/regeneration processes. The total mass loss ranged between 28.6 and 45.6%. The sludge with higher mass loss was DWTR6 and the lower was DWTR2. These values may suggest that in the DWTR regeneration/reactivation process, a significant mass would be lost, compromising the number of reactivation cycles that this material can endure. Other studies have reported similar results, Dias et al. (2021) reported a total mass loss in AC-DWTR of 27 and 34%, and Likus et al. (2021) reported a total mass loss between 29 and 39% in Fe-DWTR. While the Al-DWTR used in Martins et al. (2022) study was more stable (total mass loss slightly lower=21%). Until 105 °C, the behaviour of the DWTR was very similar. The mass loss observed until this temperature was associated with the moisture of the samples. After this temperature, it was possible to identify some differences in the thermal decomposition behaviour. For DWTR 2, 5 and 6, (see Fig. 1) it was possible to identify that the most significant mass loss takes place between 200 and 480 °C, which can be related to the degradation of the organic matter (Pellenz et al. 2023). Among these three, DWTR 6 exhibited the highest mass loss in this temperature range, indicating a potentially elevated organic matter content within this sludge. In Fig. 1 was possible to identify a mass loss between 200 and 480 °C in DWTR 1, 3, 4 and 7, however, lower when compared to the other DWTR. The most significant mass loss identified in these four DWTR happened around 750 °C, which may be related to the degradation of CaCO_3 (Bernardo et al. 2020). This is supported by the mineral analysis results (see Table SM2), as these four DWTR have the highest Ca content.

The FTIR spectra of the samples are quite similar (see Figure SM2 of the Supplementary Material). All the DWTR had bands associated with the presence of aluminium hydroxides ($3\ 430$ and $530\ \text{cm}^{-1}$) that are correlated with the coagulant used in the drinking water treatment process (Lee et al. 2020). In the FTIR spectra was also possible to verify that all the DWTR had bands related with Si compounds ($1\ 050\ \text{cm}^{-1}$) (Coates 2000; Lee et al. 2020). The presence of silicon bands may be related with the raw water composition,

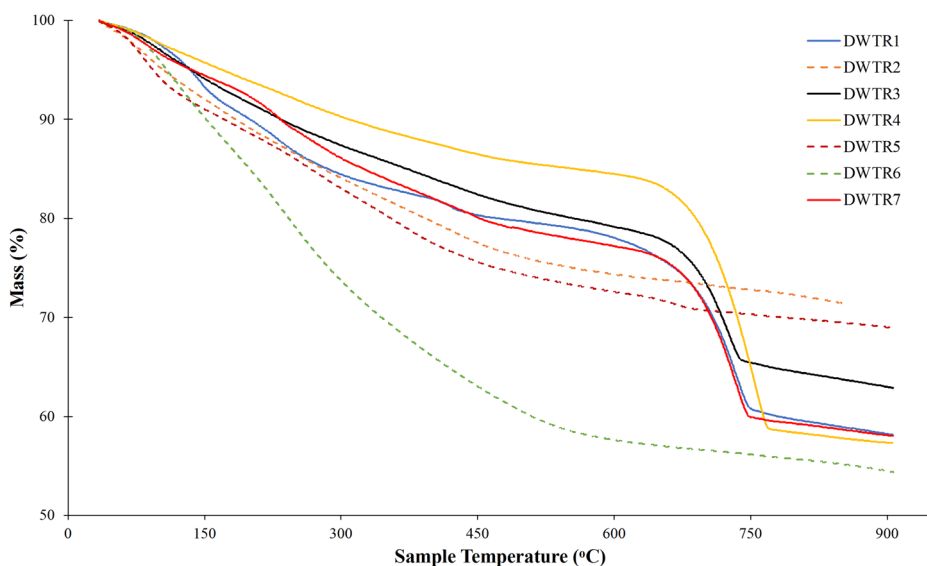


Fig. 1 Thermogravimetric analysis (TGA) of the DWTR

namely clays and riverbeds (Martins et al. 2022). In the FTIR spectra of DWTR 1, 3, 4 and 7 (Figure SM2-a) it was possible to identify bands at 875 cm^{-1} and 1490 cm^{-1} that can be linked to the carbonate ion (Coates 2000). However, this peak was not identified in the FTIR spectra of DWTR 2, 5 and 6 (Figure SM2-b), this may be explained by the differences in the Ca content. In the DWTR 1, 3, 4 and 7 was also possible to identify a peak related to S compounds, although the elemental analysis did not reveal any S content in the DWTR. In DWTR 2, 5, and 6 FTIR (Figure SM2-b) was possible to identify C-H in-plane bend at peak at 1400 cm^{-1} , respectively, that may be related to the organic matter present in the DWTR (Siswoyo et al. 2014). In these three DWTR was also possible to identify a peak around 1700 cm^{-1} associated with the presence of carboxylic acid, which is confirmed by the acidic nature of these DWTR (see pH_{PZC} in Table 1).

The SEM-EDS results of DWTR 3 (see the top of Fig. 2) showed the heterogeneity of this material since it was possible to identify particles with regular surfaces (zone E) in which the carbon was element more representative (Fig. 2 (bottom – E zone), suggesting that may be a particle of AC. Siswoyo et al. (2014) also identified particles of AC in the SEM images of the DWTR. In the SEM image was also possible to identify large particles with roughness (zone D) with high content of oxygen and Al. This type of particle was also identified by Martins et al. (2022). This heterogeneity was observed in all the seven DWTR (Figure SM3, Figure SM4, Figure SM5, Figure SM6, Figure SM7 and Figure SM8 of the

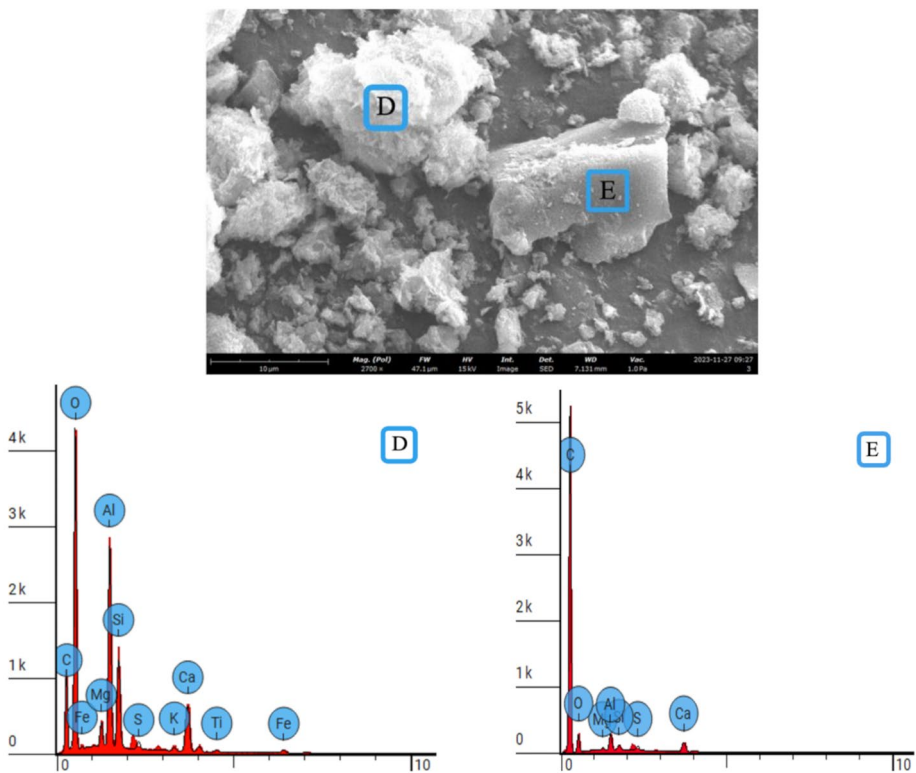


Fig. 2 DWTR3 SEM images with magnification $2\ 700\times$ (top) and EDS spectra of the selected zones (bottom)

Supplementary Material). EDS spectra reveal the presence of both Si and S elements on the particle's surface, confirming the results of the FTIR analysis.

The results of the leaching assay (see Table SM3 of the Supplementary Material) show that the pH variation is related to the DWTR pH_{PZC} since the DWTR with alkaline properties led to an increase in the pH of the solution due to the release of OH^- into the solution, whereas the DWTR with acidic properties led to a decrease in the pH of the solution due to the release of H^+ ions (Muisa et al. 2020). In the leaching samples was also possible to observe an increase of the conductivity between 611 and 1 793 $\mu\text{S}/\text{cm}$ at a temperature of 20° C. The increase observed may be related to the amount of Cl^- that was released in the leaching. The Cl^- released may be associated with the coagulants used in the DWTP, namely polyaluminium chloride. Among the chemical species analyzed, Na was the one with higher relative mobility, followed by Ca. While the Al relative mobility was lower than 1% for almost all the DWTR, except for DWTR 1 which achieved a relative mobility of 3.9%, which means a leaching of 2.85 $\mu\text{g}/\text{g}$. Considering the conditions of the adsorption test (0.1 g of DWTR and 200 mL of solution), the Al content released by the studied DWTR would range between 0.0 and 1.4 $\mu\text{g}/\text{L}$. This range is below the parametric value for water quality intended for human consumption stabilised by the Directive (EU) 2020/2184 of the European Parliament and of The Council (2020).

3.2 Phosphate Adsorption Experiment

Given that DWTR demonstrates a high capacity for phosphate adsorption, this study used a phosphate concentration higher than typical levels found in wastewater to achieve sludge saturation. This strategy enables the evaluation of which properties most effectively indicate the most suitable sludge for use as a phosphorus adsorbent. The PO_4^{3-} adsorption results showed that all DWTR, except DWTR 6, were able to adsorb P O_4^{3-} , however, there were differences between the amount of PO_4^{3-} adsorbed (see Table 3). DWTS 6 only achieved an uptake capacity of 0.2 mg/g, this may be due to its low V_{total} and S_{BET} (Table 2) since the textural properties are known to influence the PO_4^{3-} adsorption onto the sludge (Lee et al. 2020). The other DWTR results were in line with the ones reported in the literature. Lee et al. (2020) for the same 50 mg/L of PO_4^{3-} but with a higher DWTR dose (1 g in 50 mL) achieved an uptake capacity lower than 1.5 mg/g. While Hou et al. (2018), who studied four DWTR as an adsorbent of phosphorous attained slightly lower uptake capacities (2.06–6.06 mg/g) than the ones obtained for all the DWTR, except DWTR 6.

Zhao and Yang (2010) reported higher values of uptake capacity for the phosphorus, namely 13.1 and 14.3 mg/g at pH 7. However, at lower pH levels, the reported adsorption capacities were higher. For instance, at pH 4, the values were 22.4 and 20.1 mg/g for each

Table 3 Phosphate adsorption capacity and removal results achieved by each DWTR

Sample	q (mg/g) ¹	Removal efficiency (%)
DWTR1	44.11 ± 0.02	99.51 ± 0.11
DWTR2	6.72 ± 0.05	15.22 ± 0.17
DWTR3	25.88 ± 0.18	58.39 ± 0.39
DWTR4	8.45 ± 0.87	19.17 ± 2.01
DWTR5	9.01 ± 0.62	20.37 ± 1.42
DWTR6	0.22 ± 0.30	0.29 ± 0.58
DWTR7	9.45 ± 0.53	21.39 ± 1.01

¹mean ± standard deviation

DWTR studied by Zhao and Yang (2010). This phenomenon can be attributed to the fact that phosphorus adsorption tends to increase with decreasing pH. Despite this, the results obtained at pH 4 in the Zhao and Yang (2010) study were lower than the values obtained for DWTR 1 and 3. Interestingly, DWTR 1, which has a lower S_{BET} than the DWTR studied by Zhao and Yang (approximately $50 \text{ m}^2/\text{g}$), exhibited higher adsorption capacity. Nguyen et al. (2022a) conducted a study on phosphate adsorption in sludge with a specific surface area (S_{BET}) of $237.9 \text{ m}^2/\text{g}$, which is higher than that of the sludge analyzed in this study (as shown in Table 2). Despite this difference, Nguyen et al. reported an adsorption capacity of 6.7 mg/g . This suggests that S_{BET} may not be the sole determining factor for phosphorus adsorption. In Rahmati et al. (2022) work where it was used 0.4 mg of DWTR in 50 mL of a solution with 50 mg/L of phosphorus it was achieved an uptake capacity lower than 6 mg/g .

According to Rahmati et al. (2022), the Al content can be used to assess the PO_4^{3-} adsorption onto DWTR. Similarly, Nguyen et al. (2022a) reported that the Al content can be used as a predictor of the adsorption capacity. However, even though DWTR samples 5 and 6 have higher Al content, they did not exhibit the highest adsorption capacity values. This suggests that the adsorption of PO_4^{3-} onto DWTR may not be solely influenced by the aluminium content, as other parameters may also play a role. DWTR 1 and 3 achieved the higher uptake capacity values, although they do not have the highest Al content, this may be related to the Ca content, which influences the PO_4^{3-} adsorption (Bernardo et al. 2020). It seems that the pH_{PZC} is another parameter that influences the PO_4^{3-} adsorption, since the higher uptake capacity results were achieved in DWTR that were more alkaline. This could be associated with the prevalence of positive charges in the DWTR surface, that promote the adsorption of anionic forms such as PO_4^{3-} .

3.3 Predictor Selection and Modelling

The correlation between the q achieved and some of the DWTR properties showed that the pH_{PZC} is one of the properties that most influence the adsorption of PO_4^{3-} (see Table SM4). The C, O and N content also had correlation factors higher than 0.8, which could suggest that these elements could be considered factors that influence adsorption. However, since the content of these three elements was very similar between the DWTR is not possible to conclude their influence. Since the PO_4^{3-} adsorption capacity was strongly correlated with the pH_{PZC} , a linear model was constructed to determine how effectively this parameter could be used to estimate the uptake capacity of PO_4^{3-} (Table 4).

Since $p\text{-value} < 0.05$ it was possible to assume that this model (Eq. (4)) can be used to estimate around 73.5% of the PO_4^{3-} adsorption capacity based on the DWTR's pH_{PZC} . The estimated coefficient for the pH_{PZC} was significant and positive which suggests that the higher the pH_{PZC} the higher will be the q obtained.

Table 4 Linear model for PO_4^{3-} adsorption capacity based on the dwtr's pH_{PZC}

Source	Degrees of Freedom	Adjusted R	F-statistic	p -value
Model	19	0.7351	56.5	<0.0001
Coefficients	Estimate	Standard Error	t Ratio	Pr (< t)
Intercept	-24.6645	5.4934	-4.490	0.000251
pH_{PZC}	5.2058	0.6926	7.517	<0.0001

Table 5 Model validation: comparison between the experimental adsorption capacity and the adsorption capacity estimated with the models

pH_{PZC}	$q_{\text{experimental}}$ (mg/g) ¹	q_{model} (mg/g)
11.29	32.24 ± 0.10	34.11
7.46	16.14 ± 2.57	14.2

¹mean ± standard deviation

$$q_{\text{PO}_4^{3-}} = 5.2058 \times \text{pH}_{\text{PZC}} - 24.6645 \quad (4)$$

The use of pH_{PZC} as a predictor of phosphate adsorption capacity was validated using the DWTR characterised in the study carried out by Rita et al. (2021). Table 5 presents a comparison between the experimental results for DWTR from the study by Rita et al. (2021) and the values predicted by the linear model. Comparing the experimental results with the predicted values reveals a difference of approximately 2 mg/g between them. These results show that pH_{PZC} can be used to predict the DWTR adsorption capacity sludge can be absorbed. This means that water utilities can use this model to select the sludge with the best phosphate removal performance from a variety of DWTR.

By establishing pH_{PZC} as a reliable indicator of DWTR adsorption capacity, this research empowers water utilities to efficiently choose DWTR with superior phosphate removal capabilities. Using a key parameter will reduce the number of analyses and enable a more streamlined decision-making framework for DWTR reuse among stakeholders. However, it should be noted that although there is a correlation between pH_{PZC} and adsorption capacity, this does not mean that there are no other parameters that influence adsorption capacity.

4 Conclusion

The characterization performed in this work revealed differences in the physical and chemical properties of the studied DWTR, despite the incorporation of identical chemical reagents. This confirms that the properties of the DWTR are closely linked to the drinking water treatment process at the DWTP and the quality of the raw water. Despite their differences, nearly all DWTR demonstrated the capacity to adsorb PO_4^{3-} , although their adsorption capacities varied. Contrary to what the scientific literature suggests, it was discovered that aluminium was not the determining factor in PO_4^{3-} adsorption capacity, despite its known ability to enhance the adsorption of phosphorus forms.

The findings indicated that the higher the pH_{PZC} of the DWTR, the greater the adsorption capacity in a neutral pH solution. This may be related to the prevalence of positive charges at the DWTR surface, which promote the adsorption of the anionic PO_4^{3-} . Therefore, pH_{PZC} could be used for forecasting the DWTR that are most suitable to adsorb PO_4^{3-} . The use of the pH_{PZC} enabled the prediction of the adsorption capacity of two DWTR with only a 2 mg/g deviation from the experimental results of the validation experiment. The findings suggest that pH_{PZC} serves as a useful tool for predicting the adsorption capacity of DWTR. As a result, water utilities can use this model to select the most suitable DWTR for phosphate removal, thereby minimizing the number of analyses required for DWTR selection. This approach streamlines the decision-making process for stakeholders regarding DWTR reuse.

The main limitation of this study was the inability to incorporate morphological properties (e.g. SEM results) which, although influencing adsorption, could not be quantified and were therefore not included in the model. The heterogeneity of this material is another limitation, since its properties are strongly dependent on the raw water and the treatment. Future studies should focus on analysing the key parameters for the adsorption of other compounds, namely nitrogen and pollutants of emerging concern, like antibiotics. In addition, machine learning tools should be explored to develop decision support models based on DWTR characteristics and the water properties.

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Data Availability Data will be made available on request.

Declarations

Competing Interests The authors declare no competing interests.

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