

## Article

# Analysis of Leachates in Activated Char from Textile Sources: Implications for Their Use as Adsorbents

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## Abstract

Textile waste management remains a critical environmental challenge. Valorization through thermochemical routes such as pyrolysis offers a sustainable pathway within the circular economy. In this study, carbonaceous materials were obtained from the pyrolysis of 100% cotton textile residues and subsequently activated with sodium thiosulfate ( $\text{Na}_2\text{S}_2\text{O}_3$ ). The physicochemical and leaching behaviors of the activated (CA) and non-activated (C) chars were assessed in aqueous solution under controlled pH conditions (3, 7, and 11). Activation significantly increased the specific surface area (from 90 to 975  $\text{m}^2 \text{g}^{-1}$ ). Leaching tests revealed that acidic conditions (pH = 3) enhanced the release of major elements following the order  $\text{Na} > \text{Ca} > \text{K}$  for C and  $\text{S} > \text{Ca} > \text{Na}$  for CA. Despite this, all concentrations of major and trace metals remained well below regulatory discharge limits. Anionic species ( $\text{Cl}^-$ ,  $\text{SO}_4^{2-}$ ) increased slightly after activation but also stayed within safe thresholds, and chemical oxygen demand (COD) values were low (0–9  $\text{mg O}_2 \text{L}^{-1}$ ), indicating negligible organic leaching. Overall, the findings show that the structural quality of textile-derived chars was improved by  $\text{Na}_2\text{S}_2\text{O}_3$  activation without compromising their environmental stability, validating their applicability as effective and safe adsorbents for wastewater treatment applications.

**Keywords:** textile waste; pyrolysis char; sodium thiosulfate activation; leaching; adsorbents

## 1. Introduction

The need for novel technologies that can recover textile waste and contribute to the transition from the traditional linear paradigm in favor of closed-loop, sustainable alternatives has been prompted by the increasing production of this waste [1]. In this context, a variety of recycling technologies have been investigated, and, in this context, thermochemical processes, particularly pyrolysis, have shown great potential. Pyrolysis is defined as the thermal decomposition of organic matter in the absence of oxygen [2]. Three fractions are normally formed during this thermochemical process: (i) a gaseous fraction enriched in non-condensable hydrocarbons; (ii) a condensable liquid fraction (bio-oil), and (iii) a solid carbonaceous fraction, sometimes referred as char [3], with feedstock composition and operational conditions having a significant impact on the distribution and properties of these products [4].

Due to its versatility in turning different types of waste as plastics [5], biomass [6], and, more recently, textile waste [4,7] into high-value products, this technology has attracted



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a lot of attention lately. Recently, some researchers have examined the pyrolysis-based valorization of textile waste. For example, Sina and Monsurat [8] showed how textile pyrolysis can be used in Nigeria to produce and purify petrochemical compounds. Similarly, Ciuffi et al. [9] studied the slow pyrolysis of textile waste to produce char and found that the resulting material had an interesting microporous structure (<2 nm) and a high surface area of about 319 m<sup>2</sup>/g. Other works, such as Arjona et al. [10], Xia et al. [11], and Silva et al. [12], have reported on the potential of textile waste in the production of charcoal.

Several studies have further focused on the production of activated carbons using activating agents such as KOH [13], H<sub>3</sub>PO<sub>4</sub> [14], and ZnCl<sub>2</sub> [15], obtaining highly porous materials with a high adsorption capacity for emerging pollutants and dyes. In contrast, in the present study, Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> was selected as an alternative activating agent due to its lower toxicity, reduced corrosiveness, and easier handling, aiming to balance adsorption performance and environmental safety.

Although numerous studies have evaluated the adsorption capacity of waste-derived carbons, the potential leaching of organic compounds and trace metals from these materials has been far less investigated. In this context, the investigation of leaching processes emerges as a critical component of a comprehensive environmental assessment, as the potential release of organic molecules, trace metals, or other chemical species could compromise the environmental benefits of this material by introducing uncontrolled secondary pollution [16–18]. Furthermore, although specific regulatory standards for acceptable leaching from textile-based carbons have not yet been established, the present work provides fundamental data to support future risk assessments and the development of appropriate regulatory frameworks [19–21].

For example, alkali-activated and Portland cement pervious concretes have demonstrated effective removal of multiple heavy metals from water while exhibiting limited leaching of adsorbed metals under long-term operation conditions [16]. Similarly, iron-based oxy-hydroxide adsorbents have achieved high removal efficiencies for Sb(III) in water and were classified as inert waste after leaching tests [17], highlighting the importance of post-adsorption environmental assessment. In pyrolysis-derived carbons, activation and stabilization of chars from plastic or contaminated organic wastes significantly enhanced adsorption capacity for heavy metals while reducing the leaching of organic compounds and trace elements [22]. Additionally, studies on heavy metal fate during pyrolysis show that most metals are retained in the solid fraction, though leachability depends on feedstock, temperature, and matrix properties [18], emphasizing the need for systematic leaching evaluations.

Currently, there are no specific regulations for the study of activated carbons derived from textile waste. However, standards applicable to conventional activated carbon and solid waste have been used as a reference, including tests for physical, chemical and leaching properties relevant to water treatment [23].

It should be noted that when these materials are intended for use in water treatment for human consumption, the applicable regulations establish more stringent safety criteria. For powdered activated char, Spanish standard UNE-EN 12903 [24] is used, and for granular carbon, UNE-EN 12915-1 [25] and UNE-EN 12915-2 [26] are used. The selection of these standards is based on the geographical relevance of the study, which was conducted in Spain. Moreover, as Spanish standards are aligned with European regulations, they are widely recognized in the European scientific literature, ensuring methodological consistency and comparability of the results. On the other hand, to ensure the quality and performance of activated char in a variety of applications, the American Society for Testing and Materials (ASTM) sets strict guidelines for its use and characterization [23,27].

Although these standards do not specifically address activated char derived from textile waste, they provide a useful framework for assessing material quality.

Leaching tests were carried out in compliance with the UNE-EN 12457-2 standard [28]. Both European and local classification criteria were applied, as they are complementary and ensure a robust interpretation of the potential substances that may be released into the environment under different discharge scenarios. Specifically, the European framework (Decision 2003/33/EC) addresses waste intended for landfill disposal and provides a broad technical scope and population coverage, thereby facilitating comparison with similar studies worldwide [29]. In addition, the inclusion of local regulatory criteria focused on discharges into municipal sewer systems (Municipal Wastewater Discharge Ordinance) is essential for contextualizing the findings within a practical local management framework. It should be noted that this local regulatory framework is based on Spanish water legislation, which in turn transposes the relevant European directives [30–32].

## 2. Materials and Methods

### 2.1. Methods for the Characterization of the Char

The chars were obtained by pyrolysis of textile waste (jeans waste) at 800 °C in a Nabertherm R50/250/12 tubular furnace (Lilienthal, Germany) operated under an N<sub>2</sub> atmosphere (50 L h<sup>-1</sup>). The reactor, constructed of 316 stainless steel, had an internal diameter of 4 cm and a length of 34.25 cm. The process was conducted at a heating rate of 10 °C min<sup>-1</sup> and a residence time of 90 min. The conditions have been chosen in accordance with the previous work of the authors [22,33]. Activated char was subsequently prepared by chemical impregnation with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub>. To do this, 15 g of denim was cut, placed in a stainless-steel crucible, and impregnated with a solution containing 15 g of thiosulfate dissolved in 20 mL of water. The impregnated fabric was placed in an oven at 120 °C for 24 h to evaporate the water. The impregnated and dried denim was subjected to chemical activation at 800 °C, under the same conditions as for the preparation of the C, as described above.

The materials were characterized by Fourier Transform Infrared Spectroscopy (FTIR) using a Perkin Elmer Spectrum 65 spectrometer (Shelton, CT, USA), equipped with an Attenuated Total Reflectance (ATR) accessory, operating within the spectral range of 4000 to 550 cm<sup>-1</sup>.

The apparent density (compacted and uncompacted) was determined in accordance with the Spanish standard UNE-EN 12902 [34].

pH was measured using a CRISON digital potentiometer, model BasiC 20 (Hospitalet de Llobregat, Spain), previously calibrated with buffer solutions. Conductivity was measured using a CRISON multimeter, model MM 40 (Hospitalet de Llobregat, Spain).

The specific surface area ( $S_{\text{BET}}$ ) was determined from nitrogen adsorption–desorption isotherms measured at 77 K using a physisorption gas station 200 Sync from 3P-Instruments® (Odelzhausen, Germany) and applying the Brunauer–Emmett–Teller (BET) standardized method. Prior to the analysis, the sample was degassed at 100 °C for 24 h. The BET method was applied using a relative pressure ( $P/P_0$ ) range from 0 to 1.

In addition, the proximate analysis, moisture, volatile matter, fixed carbon, and ash In addition, the proximate analysis—moisture, volatile matter, fixed carbon, and ash content—was determined by thermogravimetric analysis (TGA) using a PerkinElmer thermobalance, model STA 6000 (Shelton, CT, USA), following these steps: (i) moisture content was determined as the mass loss after heating the sample at 105 °C until constant weight, in accordance with UNE-EN ISO 18134-2:2024 [35]; (ii) volatile matter was quantified as the mass loss occurring after heating the dried sample under an inert atmosphere, according to UNE-EN ISO 18123:2024 [36]; (iii) ash content was determined as the residue remaining

after complete combustion of the sample in air, following UNE-EN ISO 18122:2023 [37]; and (iv) fixed carbon was calculated by difference.

The major and minor elements in the dry material were quantified using an inductively coupled plasma optical emission spectrometer (ICP-OES) and an inductively coupled plasma mass spectrometer (ICP-MS), Optima 8300 and Nexion 300D, both of Perkin Elmer (Shelton, CT, USA), after digestion according to UNE-EN ISO 54321:2021 [38].

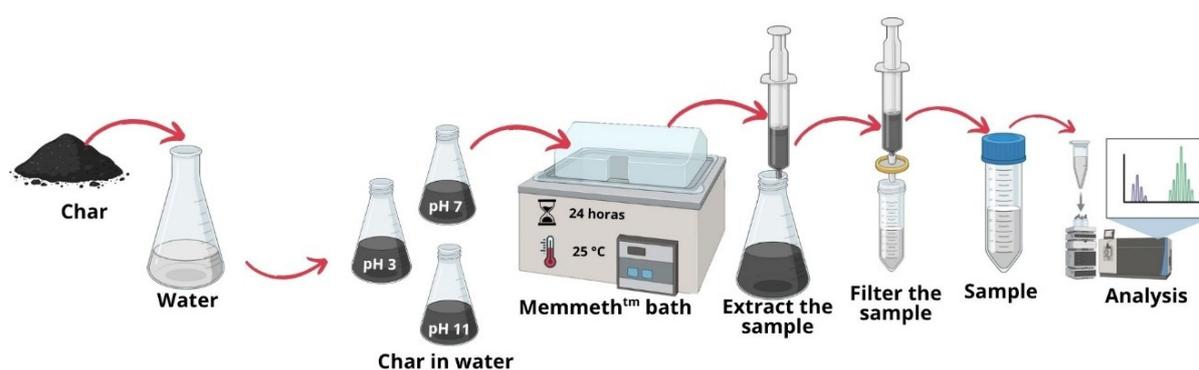
Finally, elemental analysis (C, H, N, and S) was performed in compliance with UNE-EN ISO 16948:2015 [39] using a ThermoScientific CHNS analyzer model Flash 2000 (Waltham, MA, USA), while oxygen content was estimated by difference.

## 2.2. Leaching Tests

Leaching tests were performed on C and CA materials. This study was based on a protocol previously developed by our research group, with specific adaptations for the current objectives [22]. The experiments were carried out in 100 mL Erlenmeyer flasks, in which the samples were suspended in ultrapure Milli-Q water (resistance 18.2 M $\Omega$ ·cm) at a solid–liquid ratio of 2 g L<sup>-1</sup>. Although UNE-EN 12457-2 specifies a liquid-to-solid ratio of 10 L kg<sup>-1</sup>, a lower solid loading (2 g L<sup>-1</sup>) was intentionally used in this study to better approximate realistic operating conditions in wastewater treatment applications rather than landfill disposal scenarios.

The tests were performed at three representative pH values: acidic (pH = 3), neutral (pH = 7), and alkaline (pH = 11), adjusted by adding HCl and NaOH solutions. The samples were coded as follows: non-activated carbon (C) at the three pH values (C-3, C-7, C-11) and carbon activated with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> (CA) at the corresponding pH conditions (CA-3, CA-7, CA-11). These pH values were selected to cover neutral conditions representative of most natural water environments, together with acidic and alkaline extremes to evaluate the leaching behavior under worst-case scenarios.

Suspensions were continuously stirred at 24 ± 1 °C for 24 h to ensure equilibrium, using a Memmert™ thermostatic bath (Schwabach, Germany). After this period, samples were collected and filtered through 0.45 µm PVDF syringe filters, as shown in Figure 1. All experiments were performed in triplicate, and results are reported as the mean of the three measurements. Relative standard deviations were below 5% in all cases.



**Figure 1.** Experimental setup for leaching tests of activated and non-activated chars.

## 2.3. Analysis of Leachate Composition

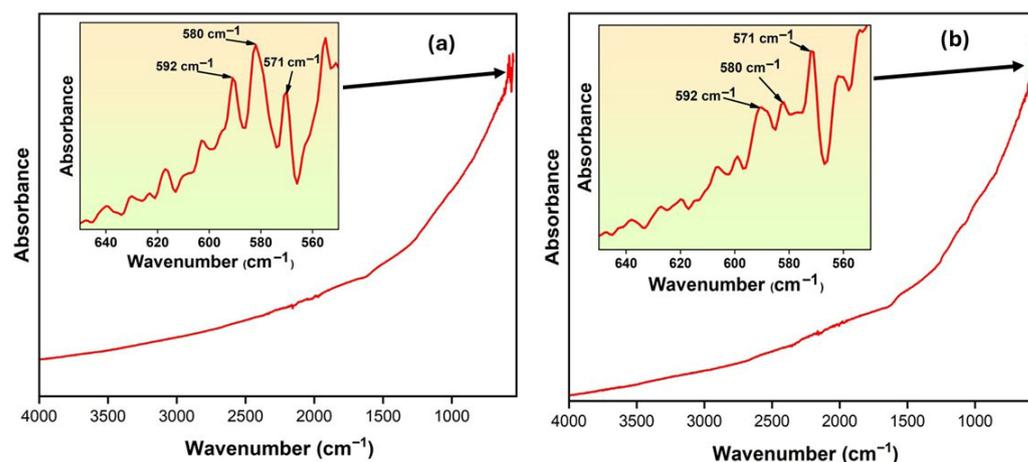
Chemical oxygen demand (COD) was measured using a commercial kit (HI93754X-25) from the commercial company HANNA instruments (Woonsocket, RI, USA). The major and minor elements were quantified using an inductively coupled plasma optical emission spectrometer (ICP-OES) and an inductively coupled plasma mass spectrometer (ICP-MS), Optima 8300 and Nexion 300D, both from Perkin Elmer (Shelton, CT, USA), and

ionic species were quantified using a 940 Professional IC Vario ion chromatograph from Metrohm (Herisau, Switzerland).

### 3. Results and Discussion

#### 3.1. Characterization of Char

Figure 2 shows the FTIR spectra of C (Figure 2a) and CA (Figure 2b). No significant bands are observed in either material in the range between 4000 and 650  $\text{cm}^{-1}$ , with only the signals in the low-frequency region (650–550  $\text{cm}^{-1}$ ) being more evident. In the C samples, two peaks are identified in the range of 600 to 580  $\text{cm}^{-1}$ , which are associated with out-of-plane bending vibrations of C–H bonds in aromatic systems or with the presence of inorganic traces [40].



**Figure 2.** FTIR spectra of char (a) and activated char with  $\text{Na}_2\text{S}_2\text{O}_3$  (b).

In contrast, in CA, these peaks remain, although with lower intensity, and greater absorbance is observed at 571  $\text{cm}^{-1}$ , indicating the presence of C–O–C functional groups and metals, in accordance with the report by Patel et al. (2021) [41]. The general absence of bands in both spectra is related to the high production temperature of the charcoal. At 800 °C, the pyrolysis reaction is practically complete, leading to a significant reduction in the functional groups present on the surface of the material [42].

The results of the analysis of the physicochemical characteristics of C and CA are summarized in Table 1. The apparent density of CA was higher than that of C in both non-compacted (128.0 vs. 84.1  $\text{kg m}^{-3}$ ) and compacted (176.8 vs. 141.7  $\text{kg m}^{-3}$ ) forms, suggesting that partial densification of the carbon matrix and structural rearrangement were encouraged by the activation process. March and Rodríguez [43] reported densities of powdered and granular carbon and indicated that this is important for determining the degree of carbon required for an existing system. This directly implies its importance in logistical considerations, as it directly influences the transport and storage of the material. Higher compacted density means that a greater mass of products can be contained in a smaller volume, thus optimizing costs and operational efficiency.

In terms of pH, both materials have a similar slightly basic pH (7.9) when measured in aqueous suspension, indicating that activation does not substantially alter the surface acidity under these conditions. However, the electrical conductivity of the suspension containing CA (198.8  $\mu\text{S cm}^{-1}$ ) was approximately 13 times higher than that of the non-activated char (15.0  $\mu\text{S cm}^{-1}$ ), reflecting the release of a higher amount of soluble ionic species or exchangeable surface functionalities introduced during chemical activation.

The  $S_{\text{BET}}$  of material C is approximately 90  $\text{m}^2 \text{g}^{-1}$ , while for material CA, this value increases to 974  $\text{m}^2 \text{g}^{-1}$ . Although the use of this activating agent has been little studied,

the results obtained demonstrate its high potential for generating highly porous structures. This is also reflected in the ratio of microporous volume to total volume, which increases from 19.8% in C to 68.0% in CA. These data indicate that chemical activation significantly improves the microporosity of the material, making it very promising for applications in contaminant adsorption.

**Table 1.** Physicochemical characteristics of char and activated char.

| Characteristic                                     | Material        |       |       |
|--|-----------------|-------|-------|
|  | C               | CA    |       |
| Apparent density (kg m <sup>-3</sup> )             | Non-compacted   | 84.1  | 128.0 |
|  | Compacted       | 141.7 | 176.8 |
| pH   |                 | 7.9   | 7.9   |
| Conductivity (μS cm <sup>-1</sup> )                |                 | 15.0  | 198.8 |
| S <sub>BET</sub> (m <sup>2</sup> g <sup>-1</sup> ) |                 | 90    | 974   |
| V <sub>MP</sub> (cm <sup>3</sup> g <sup>-1</sup> ) |                 | 0.019 | 0.418 |
| V <sub>T</sub> (cm <sup>3</sup> g <sup>-1</sup> )  |                 | 0.096 | 0.608 |
| V <sub>MP</sub> /V <sub>T</sub> (%)                |                 | 19.8  | 68.0  |
| Proximate analysis (wt.%, wet basis)               |                 |       |       |
|  | Moisture        | 5.97  | 7.43  |
|  | Volatile matter | 57.72 | 22.94 |
|  | Fixed carbon    | 33.94 | 51.79 |
|  | Ash             | 2.37  | 17.85 |
| Elemental analysis (wt.%, dry basis)               |                 |       |       |
|  | Carbon          | 77.5  | 75.9  |
|  | Hydrogen        | 0.4   | 0.2   |
|  | Nitrogen        | 0.9   | 1.0   |
|  | Sulfur          | 0.0   | 3.7   |
|  | Oxygen *        | 21.2  | 19.2  |

S<sub>BET</sub>, total surface area by BET method; V<sub>T</sub>, total pore volume from N<sub>2</sub> uptake at P/P<sub>0</sub>~0.99; V<sub>MP</sub>, micropore volume by *t*-plot method. \* By difference after ash removal.

Regarding the proximate analysis, fixed carbon increased (from 33.9% to 51.8%) while volatile matter decreased (from 57.7% to 22.9%), reflecting enhanced carbonization of the char matrix. The high ash content of the CA sample can be attributed to inorganic species introduced during chemical activation. However, elemental analysis showed minor variations in carbon, hydrogen, and nitrogen, while sulfur was notably increased in the CA sample, consistent with the use of Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> as an activating agent. Under the high-temperature activation conditions (800 °C), thiosulfate is thermally unstable and can decompose into a mixture of oxidized and reduced sulfur species.

Tables 2 and 3 show the results of the ICP-OES and ICP-MS elemental analyses of major and minor metals on a dry basis for C and CA. Also, it should be noted that during wet digestion methods for elemental analysis, sample losses may occur due to adsorption, volatilization, or co-extraction, and the extraction process may depend on the type of matrix in which the elements are found [44].

As shown in Table 2, material C is mainly composed of Na and Ca, followed by K and Fe. After activation (CA), Na and Ca are followed by K and Fe. After activation (CA), Na and Ca remain the dominant elements, but noticeable changes in elemental composition are observed. In particular, S, which is not detected in C, becomes one of the major elements in CA, reaching 13.10 g kg<sup>-1</sup>. This increase indicates the incorporation of S into the carbon

matrix as a consequence of the thiosulfate ( $S_2O_3^{2-}$ ) treatment. In addition, Al appears after activation, while K and Fe show moderate increases.

**Table 2.** Major metals in dry material.

| Analyzed Material | Concentration ( $g\ kg^{-1}$ of Dry Matter) |      |       |       |       |       |      |       |
|-------------------|---|------|-------|-------|-------|-------|------|-------|
|                   | Na  | K    | Al    | Ca    | P     | Mg    | Fe   | S     |
| C                 | 10.96                                       | 2.71 | <d.l. | 5.35  | <d.l. | <d.l. | 0.86 | 0.00  |
| CA                | 32.90                                       | 5.90 | 9.70  | 13.20 | <d.l. | <d.l. | 0.60 | 13.10 |

d.l.: detection limit.

**Table 3.** Minor metals in dry material.

| Analyzed Material | Concentration ( $mg\ kg^{-1}$ of Dry Matter) |       |       |       |       |       |       |       |       |       |       |       |
|-------------------|--|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|-------|
|                   | As   | Ba    | Cd    | Co    | Zn    | Cu    | Cr    | Sn    | Mn    | Hg    | Ni    | Pb    |
| C                 | 0.126  | 0.016 | 0.054 | 0.904 | 133.7 | 52.92 | 34.25 | 6.321 | 22.64 | 0.018 | 27.61 | 0.652 |
| CA                | 0.220  | 76.37 | 0.040 | 3.200 | 189.2 | 55.92 | 174.9 | 565.7 | 37.79 | 0.300 | 123.5 | 19.99 |

As for minor metals, shown in Table 3, the results indicate that the activation process significantly alters both the distribution and concentration of several metals. The presence of these metals is related to the intrinsic composition of the textile, since metals are used during the production process as catalysts, in dyes, pigments, or as finishing agents [44–47].

Ar, Ca, and Hg showed low values in both samples, with no significant variations after activation. In contrast, the activation process resulted in a substantial increase in the concentrations of several metals, particularly Ba, Sn, Pb, Cr, and Ni. Notably, Sn and Ba exhibited the most pronounced enrichment after activation, increasing from 6.32 to 565.7  $mg\ kg^{-1}$  and from 0.016 to 76.37  $mg\ kg^{-1}$ , respectively.

This increase in metal concentrations in CA may be attributed to their incorporation or retention on the char surface during treatment with  $Na_2S_2O_3$ . Thiosulfate, which exhibits both oxidizing and reducing behavior due to the presence of multiple sulfur oxoanions, can promote the formation of stable metal–sulfur complexes [48]. Combined with the high porosity developed during activation, these conditions favor metal adsorption and retention, limiting their removal during the washing steps.

Zn and Cu already presented relatively high concentrations in the raw char (133.7 and 52.9  $mg\ kg^{-1}$ , respectively), suggesting enrichment during pyrolysis because of organic matter volatilization and the concentration of non-volatile elements in the solid residue. After activation, both metals showed a moderate increase, indicating that, rather than being leached, they were partially retained or further concentrated in the activated char matrix [49,50].

Given the structural differences after activation, leaching tests were performed to evaluate chemical stability and the main results are provided in the following sections.

### 3.2. Characterization of Water-Extractable Substances

#### 3.2.1. Major and Minor Metals

Table 4 summarizes the concentrations of the main leached elements from both C and CA material under acidic (pH = 3), neutral (pH = 7), and alkaline (pH = 11) conditions, together with the regulatory limits established by the Granada municipal ordinance whose objective is to regulate the conditions to which wastewater discharges into the municipal sewerage network must comply, with the aim of protecting water resources, preserving the environment, and ensuring public health [30].

**Table 4.** Concentrations of major metals leached from C and CA, compared with regulatory limits.

| Concentration<br>(mg L <sup>-1</sup> ) | Sample |      |      |      |      |       | Limit Values (mg L <sup>-1</sup> )<br>Municipal<br>Ordinance |
|--|--------|------|------|------|------|-------|--|
|  | C-3    | C-7  | C-11 | CA-3 | CA-7 | CA-11 |  |
| Al                                     | 1.50   | 1.50 | 1.42 | 1.43 | 2.74 | 3.13  | 20   |
| Ca                                     | 2.37   | 2.05 | 1.48 | 8.95 | 7.30 | 4.19  | ---  |
| Fe                                     | 1.11   | 1.13 | 1.11 | 1.10 | 1.11 | 1.11  | 15   |
| K                                      | 2.11   | 2.12 | 2.11 | 2.29 | 2.29 | 2.44  | ---  |
| Mg                                     | 1.18   | 1.18 | 1.19 | 1.18 | 1.18 | 1.18  | ---  |
| Na                                     | 3.53   | 2.60 | 2.66 | 8.66 | 7.32 | 7.32  | ---  |
| P                                      | 1.48   | 1.49 | 1.48 | 1.48 | 1.48 | 1.48  | ---  |
| S                                      | 0.00   | 0.00 | 0.00 | 17.3 | 5.76 | 7.73  | ---  |

Unregulated (---).

A clear influence of pH on the leaching behavior of major elements was observed for both materials. In general, acidic conditions (pH = 3) promoted higher metal release, particularly Na and Ca, while neutral and alkaline conditions reduced metal mobility. This pattern agrees with the protonation of surface functional groups that promotes electrostatic repulsion of cationic species, enhancing their desorption from the solid matrix.

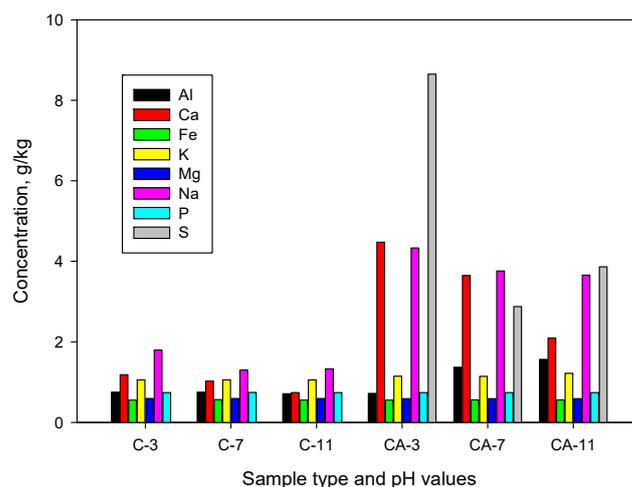
Activation with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> significantly modified the leaching behavior of several major elements. Ca exhibited the most pronounced increase after activation, particularly under acidic conditions, where its concentration rose from 2.37 mg L<sup>-1</sup> in the C to 8.95 mg L<sup>-1</sup> in the CA at pH = 3.

Na concentrations in the leachates increased consistently after activation across all pH values, reaching 8.66 mg L<sup>-1</sup> at pH = 3 in CA samples compared to 3.53 mg L<sup>-1</sup> in the C. This behavior reflects the incorporation of Na-containing species during chemical activation with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> and their subsequent partial release into the aqueous phase during leaching. In contrast, iron exhibited negligible variations between activated and non-activated samples under all pH conditions, with concentrations remaining close to 1.1 mg L<sup>-1</sup>, indicating strong retention within the carbon matrix and limited sensitivity to both activation and pH changes.

Sulfur was detected exclusively in the leachates of the activated char, with concentrations ranging from 5.76 to 17.3 mg L<sup>-1</sup> depending on pH, confirming the successful incorporation of sulfur-containing species during the activation process. Despite this increase, sulfur concentrations remained well below any applicable regulatory thresholds.

Overall, the concentrations of all major elements in the leachates remained well below the discharge limits established by the Granada municipal ordinance (20 mg L<sup>-1</sup> for Al and 15 mg L<sup>-1</sup> for Fe). Although regulatory limits are not defined for other major elements in the referenced standards, the measured concentrations were low and comparable to or lower than those reported for other environmentally safe carbonaceous materials [51,52], indicating no significant risk of secondary contamination.

Based on the data obtained in Table 4, at three pH values, expressed in mg L<sup>-1</sup>, and considering the char: liquid ratio of 2 g L<sup>-1</sup>, the corresponding conversion was performed to obtain the results in g kg<sup>-1</sup> (Figure 3). Thus, the final values represent the amount of metal released per unit mass of char. It is observed that at pH = 3, metal release is greater in both non-activated (C) and thiosulfate-activated char (CA), with pH = 7 being the optimum for working with this type of material.



**Figure 3.** Concentrations (g/kg) of major metals leached from C and CA, at the three pH levels studied.

Table 5 lists the minor elements detected in the leachates. Most trace metals were found at very low concentrations, far below the maximum permissible levels established by the Granada ordinance and UNE-EN standards. However, compared to neutral or alkaline environments, some metals, especially Ba, Zn, and Ni, exhibited comparatively increased leaching under acidic conditions (pH 3). For instance, Ni significantly rose to  $244.13 \mu\text{g L}^{-1}$  in CA-3, while Ba and Zn reached  $70.73 \mu\text{g L}^{-1}$  and  $104.57 \mu\text{g L}^{-1}$  in C-3. The slightly higher Ni concentration in CA-3 may result from complexation of  $\text{Ni}^{2+}$  with thiosulfate ligands introduced during activation, which can form soluble metal–thiosulfate complexes [53]. Conversely, the reduced leaching of Pb and Cu in CA samples compared with C indicates the possible formation of less soluble metal–sulfur species, which improve immobilization.

**Table 5.** Concentrations of minor metals leached from C and CA, compared with regulatory limits.

| Concentration ( $\mu\text{g/L}$ ) | Sample |       |       |        |       |       | Limit Values ( $\mu\text{g L}^{-1}$ ) |                |
|-----------------------------------|--------|-------|-------|--------|-------|-------|---------------------------------------|----------------|
|                                   | C-3    | C-7   | C-11  | CA-3   | CA-7  | CA-11 | Municipal Ordinance                   | UNE-EN-12915-1 |
| As                                | 0.41   | 0.05  | 0.03  | 0.41   | 0.08  | 0.11  | 1000                                  | 10             |
| Ba                                | 70.73  | 51.82 | 52.50 | 100.30 | 77.77 | 29.22 | 20,000                                | ---            |
| Cd                                | 0.06   | 0.00  | 0.00  | 0.06   | 0.00  | 0.00  | 500                                   | 0.5            |
| Co                                | 0.93   | 0.06  | 0.00  | 4.89   | 0.12  | 0.00  | 200                                   | ---            |
| Zn                                | 104.57 | 26.78 | 4.65  | 222.86 | 15.89 | 0.97  | 10,000                                | ---            |
| Cu                                | 14.44  | 0.00  | 0.00  | 4.39   | 2.31  | 0.07  | 3000                                  | ---            |
| Cr                                | 5.99   | 1.33  | 1.65  | 17.07  | 1.81  | 1.99  | 3000                                  | 5              |
| Sn                                | 0.00   | 0.00  | 0.00  | 0.00   | 0.00  | 0.00  | 2000                                  | ---            |
| Mn                                | 15.33  | 2.47  | 0.00  | 36.81  | 1.18  | 0.00  | 2000                                  | ---            |
| Hg                                | 0.00   | 0.00  | 0.00  | 0.00   | 0.00  | 0.00  | 100                                   | 0.3            |
| Ni                                | 0.00   | 0.00  | 0.00  | 244.13 | 0.00  | 0.00  | 4000                                  | 15             |
| Pb                                | 12.67  | 0.19  | 0.10  | 2.23   | 0.00  | 0.00  | 1000                                  | 5              |

Unregulated (---).

Although the acidic medium improves the dissolution of metal species weakly bound to the carbon surface [54], all the metals detected are below regulatory limits, confirming that the leachates from both carbons can be classified as environmentally safe.

The values shown in Figure 4 were obtained using the same procedure as for Figure 3. Similarly, the results show that at pH = 3, there is a greater release of metals, suggesting that acidic conditions favor the desorption of all metals in the char. With these results, solids can be classified as inert waste according to the leaching criteria established in Decision 2003/33/EC [29], since the concentrations of metals released are within the permitted values.

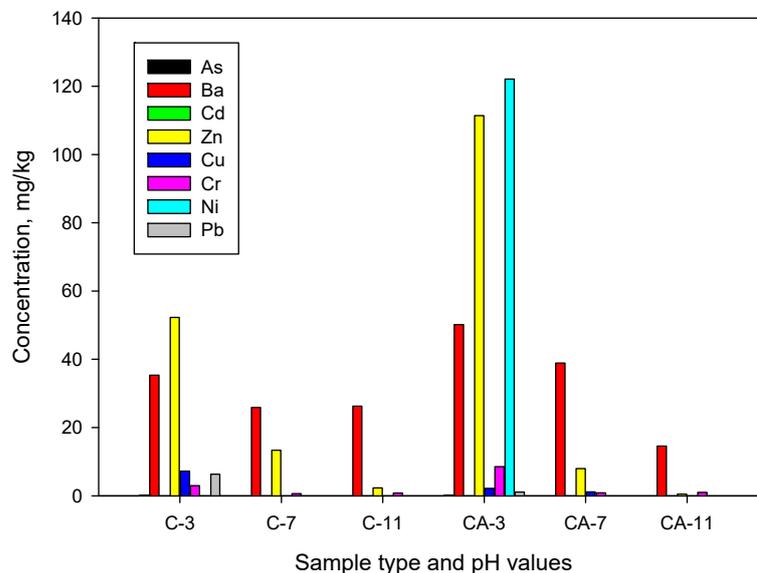


Figure 4. Concentrations (mg/kg) of minor metals leached from C and CA, at the three pH levels studied.

Figures 5 and 6 represent the percentage of metal removed from the original material. In non-activated char (Figure 5a), Na and Fe are the metals showing the highest leaching under acidic conditions, reaching values of up to 61.70% and 63.97%, respectively. Potassium also exhibits significant leaching, with approximately 40% removed regardless of the pH. In contrast, for activated char (Figure 5b), the highest leaching percentage corresponds to Fe, reaching up to 92% independently of pH, followed by sulfur, which shows a leaching percentage of 66.3% under acidic conditions. It should be noted that Figure 5 includes only those metals that could be quantified according to the results presented in Table 2.

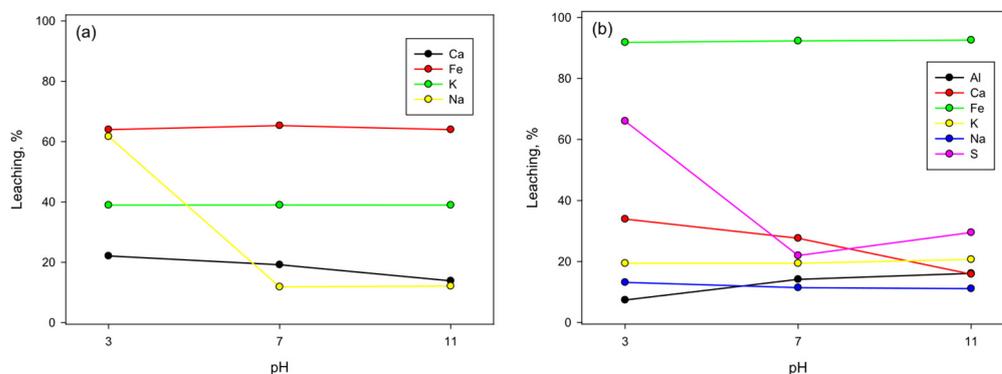
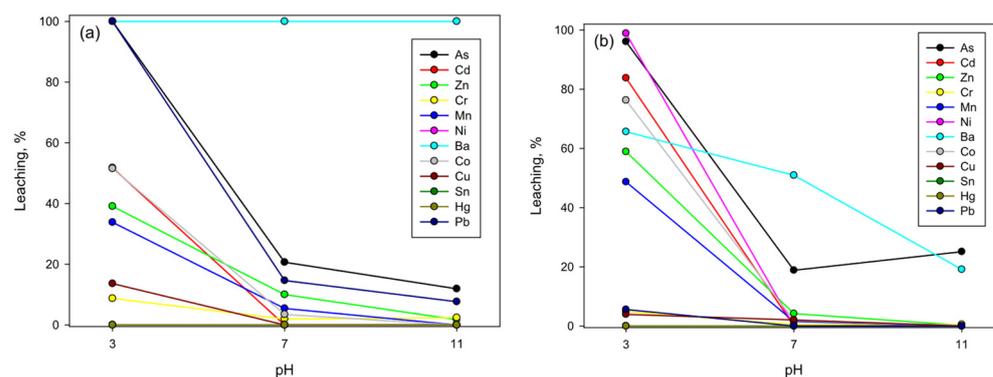


Figure 5. Leaching percentage of major elements, calculated with respect to their total content in the original material, for (a) C and (b) CA.

On the other hand, minor metals, present in both the non-activated (Figure 6a) and activated (Figure 6b) leachate, show a tendency to be released in greater proportions under acidic conditions.



**Figure 6.** Leaching percentage of minor elements, calculated with respect to their total content in the original material, for (a) C and (b) CA.

At pH 3, and consistent with the trends described above, non-activated char shows complete leaching of Ba, As, and Pb, as well as approximately 50% leaching of Cd and Co; however, their release decreases markedly as the pH of the medium increases. In the case of activated char, not only are these metals released, but Zn, Mn, and Ni are also leached. Nevertheless, the pH-dependent trend is similar for both materials, with metal leaching being favored under acidic conditions and decreasing as pH increases.

These results confirm that pH is a key parameter in adsorption processes that use this type of char, as it directly influences metal leaching. Therefore, to prevent the release of both major and minor metals, it is advisable to work under neutral pH conditions. For example, a pH of 6.5 encourages more ionic contact between the negatively charged solid surface and positively charged species, resulting in maximal adsorption, as proposed by Anoop Krishnan et al. [55].

Although the measured concentrations remained below regulatory limits even under these more adverse conditions, this behavior should be considered in applications involving the treatment of acidic wastewater. In adsorption processes, pH is one of the most critical operating parameters, as it governs both the speciation of contaminants and their interaction with the adsorbent. Therefore, in the case of acidic waters, adjusting the pH to values close to neutrality should be considered a preliminary step prior to the adsorption process.

In practice, the associated potential risks can be mitigated through simple operational measures, such as pH adjustment before treatment, preconditioning of the adsorbent to remove weakly bound metals, or the inclusion of an additional polishing step when required by discharge criteria [56].

### 3.2.2. Anionic Species

The concentrations of anionic species in leachates are shown in Table 6, together with the limits established by the Granada municipal ordinance [30].

Although the CA material at pH = 3 had the highest concentration of chloride and sulfates, at concentrations of  $78.93 \text{ mg L}^{-1}$  and  $85.82 \text{ mg L}^{-1}$ , it did not exceed the established limits. This result could be attributed to the nature of the original raw material, to environmental contaminants adsorbed during its preparation, or to the presence of chlorides in the soluble carbons that are released during leaching. In addition, the presence of sulfate ions supports that sulfur introduced during activation is mainly present in oxidized, water-extractable forms.

The observed trend indicates that higher concentrations of acid release a greater amount of anions [57]. When comparing the results with the limit values, none of the measured values exceeded the regulatory thresholds. This demonstrates that both non-

activated and activated char can be classified as environmentally safe solid waste under the leaching conditions evaluated.

**Table 6.** Concentrations of leached anionic species from C and CA, compared with regulatory limits.

| Anion (mg/L)                             | Sample |      |      |       |       |       | Limit (mg/L) Municipal Ordinance |
|--|--------|------|------|-------|-------|-------|----------------------------------|
|  | C-3    | C-7  | C-11 | CA-3  | CA-7  | CA-11 |                                  |
| Fluoride (F <sup>-</sup> )               | 0.051  | 0.00 | 0.00 | 0.05  | 0.04  | 0.00  | 10.0                             |
| Chloride (Cl <sup>-</sup> )              | 20.75  | 3.90 | 4.49 | 78.93 | 8.72  | 9.11  | 1500.00                          |
| Nitrate (NO <sub>3</sub> <sup>-</sup> )  | 0.83   | 0.69 | 0.96 | 0.64  | 0.62  | 0.62  | 150.00                           |
| Sulfate (SO <sub>4</sub> <sup>2-</sup> ) | 3.07   | 2.95 | 2.93 | 85.82 | 52.17 | 52.75 | 750.00                           |

From an application perspective, the long-term stability of anionic species under repeated adsorption–desorption cycles is an important consideration, especially for chloride and sulfate anions and acidic pH values. Although cyclic stability tests were not included in this study, stability can be further improved by pre-washing or conditioning steps to remove loosely bound species and by operating close to neutral pH.

It should be noted that anion analysis was not performed for the original samples (C and CA) due to disaggregation of the material during leaching, which artificially incorporated anions into the solution. Therefore, the leaching percentage of anions, calculated with respect to their total content in the original material, cannot be calculated.

### 3.2.3. Chemical Oxygen Demand (COD)

Table 7 provides a summary of the leachates' chemical oxygen demand (COD) values of the leachates are summarized in. The observed COD levels, which range from 0 to 9 mg O<sub>2</sub> L<sup>-1</sup>, are often quite low, suggesting that the carbonaceous materials have released very little soluble organic molecules.

**Table 7.** COD from char (C) and activated char with Na<sub>2</sub>S<sub>2</sub>O<sub>3</sub> (CA).

| Sample                                   | C-3 | C-7 | C-11 | CA-3 | CA-7 | CA-11 |
|--|-----|-----|------|------|------|-------|
| COD (mg O <sub>2</sub> L <sup>-1</sup> ) | 4   | 0   | 8    | 9    | 0    | 7     |

Under neutral (pH = 7) conditions, the COD for the non-activated char was negligible, and under acidic and alkaline conditions, it only slightly rose. Similar trends were seen in the activated char, which had low COD values. Probably, the partial solubilization of organic matter under extreme pH, which increases the reactivity and dissolution of small carbonaceous or residual organic fractions, is responsible for this slight rise at acidic and basic pHs.

The COD values found here are far lower than those found in normal leachates from other chars. Although COD in char leachates is generally reported to be low, leachates from char residues produced through the pyrolysis of various wastes often exhibit higher COD levels, ranging from hundreds to thousands mg O<sub>2</sub> L<sup>-1</sup> [22,56]. Therefore, the materials studied here can be considered environmentally benign in terms of organic leachates.

## 4. Conclusions

Chars derived from the pyrolysis of textile waste and subsequently activated with  $\text{Na}_2\text{S}_2\text{O}_3$  showed marked changes in physicochemical properties. Chemical activation increased the specific surface area from 90 to 974  $\text{m}^2 \text{g}^{-1}$  and raised the microporous volume fraction from 19.8% to 68.0%, confirming the strong pore-developing effect of thiosulfate treatment. Fixed carbon content increased from 33.9% to 51.8%, while volatile matter decreased from 57.7% to 22.9%, indicating enhanced carbonization and structural stabilization. Apparent density also increased, which is favorable for handling and packing performance.

Leaching behavior was strongly dependent on pH values. Under acidic conditions (pH = 3), the highest release of major elements was observed, with maximum concentrations of  $\text{Na} = 8.66 \text{ mg L}^{-1}$ ,  $\text{Ca} = 8.95 \text{ mg L}^{-1}$ , and  $\text{S} = 17.3 \text{ mg L}^{-1}$  in activated chars. Even in these worst-case conditions, all major metals remained well below applicable discharge limits (e.g.,  $\text{Al} \leq 3.13 \text{ mg L}^{-1}$  vs. limit 20  $\text{mg/L}$ ;  $\text{Fe} \approx 1.1 \text{ mg L}^{-1}$  vs. limit 15  $\text{mg L}^{-1}$ ). For trace metals, the highest detected values were  $\text{Ni} = 244 \text{ } \mu\text{g L}^{-1}$ ,  $\text{Zn} = 223 \text{ } \mu\text{g L}^{-1}$ , and  $\text{Ba} = 100 \text{ } \mu\text{g L}^{-1}$ . While Zn and Ba remained below the regulatory thresholds defined by municipal and UNE-EN standards, Ni exceeded the UNE-EN-12915-1 limit (15  $\text{mg L}^{-1}$ ) under acidic conditions, indicating a potential risk only under extreme pH scenarios not representative of typical operating conditions.

Anionic species increased after activation but remained far below regulatory limits, with maximum values of  $\text{Cl}^{-} = 78.9 \text{ mg L}^{-1}$  (limit 1500  $\text{mg L}^{-1}$ ) and  $\text{SO}_4^{2-} = 85.8 \text{ mg L}^{-1}$  (limit 750  $\text{mg L}^{-1}$ ). Organic leaching was negligible, with COD values ranging only from 0 to 9  $\text{mg O}_2 \text{ L}^{-1}$ , substantially lower than typical values reported for other pyrolytic chars.

These results quantitatively confirm that the produced materials are structurally robust and environmentally stable, supporting their safe application as adsorbents in wastewater treatment.

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