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Current Perspectives on the Sustainable Remediation of Lead-Contaminated Water Using Tomato Juice By-Products

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Abstract

The large quantities of agri-food waste produced worldwide by the tomato processing industry require recovery. This study evaluated the efficiency of the by-product resulting from tomato juice preparation as an innovative biomaterial for removing lead from water. The pomace was dried and tested in two forms: raw (RT) and after extraction of soluble compounds (ET). The extracts obtained from the preparation of ET, could be reintroduced into the food industry (as colorants, etc.) according to the "zero waste" principle, but further studies are needed. No other chemical pre-treatment was applied to improve the lead-adsorption capacity. The pH influence, biosorbent dosage, kinetics and equilibrium were evaluated. Analytical methods, such as atomic absorption spectrometry, elemental chemical analysis, FTIR, scanning electron microscopy, and predictive models, were applied. The outcomes demonstrated a lead-adsorption efficiency of 99.22% for ET and 89.83% for RT, an optimum pH of 4.0 ± 0.5, and an initial solution containing 20 mg Pb^{2+}/L. The Langmuir model predicted high removal capacities: 142.18 mg/g for ET and 90.91 mg/g for RT. Both forms of tomato pomace were efficient for sustainable and cost-effective water remediation, but an improvement was noticed after the extraction of soluble components that could be valorized in other products within the circular economy.

Keywords: agri-food waste; biosorbents; tomato processing industry; heavy metals; tomato pomace; water treatment; lead decontamination; sustainability; circular economy; depollution

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1. Introduction

Heavy metal contamination is associated with harmful effects on the environment and living organisms, especially affecting humans and animals [1]. Lead is a naturally occurring element present in the Earth's crust. It is commonly found in industrial wastewater originating from mining, electrical and galvanizing sectors, steel production, manufacture of explosives, lead-based paints and gasoline, etc., but also from household activities (including plumbing corrosion, etc.). According to the National Institute of Statistics of Romania [2], in 2021, the total amount of wastewater was 1801 mil. m^3, of which over 53% was produced by household activities (957 mil. m^3) and around 47% by economic activities (844 mil. m^3), divided as follows: food industry (21.61 mil. m^3), mining industry (45.02 mil. m^3), chemical and petrochemical industry (50.43 mil. m^3), metallurgical industry

(84.65 mil. m^3), other economic activities (216.12 mil. m^3), and electricity production and distribution (426.84 mil. m^3). The mining, metallurgical and electricity industries (sectors currently involving lead) produced, in 2021, over 556 mil m^3 of wastewater that could be contaminated with lead [2].

Sulfate ($PbSO_4$), chloride ($PbCl_2$), sulfide (PbS), carbonate ($PbCO_3$), oxide (PbO), and nitrate ($Pb(NO_3)_2$) are common forms of lead (Pb). Lead nitrate is very soluble in water, and it is utilized in industry and laboratories. It is highly hazardous (H410: very toxic to aquatic life, H302 + H332: hazardous if swallowed or inhaled) [3].

From wastewater, lead can pollute drinking water sources, soil, plants and animals. The lead involuntary ingested (from contaminated food products) is harmful to human health. Due to its ongoing use in a variety of products (generating residues), and its non-biodegradability and easy accumulation in the environment, many studies investigated lead's toxic effects on the environment [4,5], water quality [5,6], and human health [4,6–9]. In humans, lead exposure is linked to mental or physical development delays, attention and learning difficulties, and kidney issues, and it can affect the reproductive system (low sperm count in men or miscarriage in women), the central nervous system (encephalopathy, edema, cerebellar damage), and the cardiovascular system (blood pressure) [4,6,10–12]. It can be found in the breast milk, and then, in the baby's body, leading to serious injuries [4,6,13,14]. Consequently, lead removal from wastewater is a critical environmental concern.

Along with the other 3000 species in the *Solanaceae* family, tomato fruits (*Solanum lycopersicum* L.) are extensively used in the food industry but also have applications in medicine and even for ornamental purposes [15]. Additionally, tomato plants are part of the circular economy, as stated by the European Green Deal 2050 [16–18]. According to EUROSTAT, the total amount of tomato fruits produced in Romania in the last 15 years, between 2010 and 2024, was 6.468 tons [19], the second position after apples. Coelho et al. [20] state that around 23.40% of tomato fruits' mass is lost in the by-products (pomace) resulting from their industrial processing. Therefore, it can be estimated that during the mentioned period, about 1513.5 tons of tomato pomace were produced in Romania. Similarly, important quantities of tomato pomace are produced in many countries all over the world. Thus, tomato pomace is a worldwide abundant by-product, produced by many food industries (including juice production), and any innovative idea for its reuse should be investigated in the context of the circular economy [21].

Adsorption is currently one of the most widely used methods for the removal of heavy metal-contaminated effluents. Activated carbon [22], zeolite [23], and silica [24] have been tested as adsorbents, but their high price turns them into an economically unviable option. Therefore, various studies sought to identify the best options for accessible and effective sorbents for cleaning ecosystems contaminated with heavy metals. Thus, many efforts focus on the use of agri-food wastes/by-products in raw or slightly processed form, such as residues from lemon, beans, artichoke shells [25–27], coffee waste [28–30], watermelon, banana, orange, mango, pineapple [31], raw and extracted apple pomace [32], raw and extracted grape marc [33], etc. The important amounts of waste generated from fruits and vegetables, especially at industrial level, can be sources of stress and pollution for the environment. Pigments and other bioactive compounds can be recovered from these by-products/wastes through various extraction methods and reintroduced into the food industry as natural colorants, antioxidants, etc., or used for other purposes. Once depleted of useful components, these by-products still can be employed as inexpensive and efficient materials for water decontamination of heavy metals, such as lead. In this regard, good lead decontamination of polluted water was obtained using beans and artichoke peels [25], coffee waste [29] citrus by-products [25,26], apple pomace [32], grape marc [33], etc.

Modern and very efficient techniques are needed to rigorously investigate the heavy metal-removal potential of different new biosorbents derived from the previously mentioned sources. Atomic absorption spectrometry (AAS) is one of the methods most frequently used to determine metals due to its sensitivity, precision, and various applications [34,35]. Scanning electron microscopy (SEM), Fourier transform infrared spectroscopy (FTIR) and elemental chemical analysis with an energy dispersive X-ray detector (EDS) are modern techniques widely employed for the characterization of innovative adsorbents [36–38].

There are very few previous publications that tested biomaterials obtained from tomato by-products as biosorbents for heavy metal adsorption from water [32,39,40], and from these, most of them focused on chemically modified tomato pomace. Vafakhah et al. [40] described the adsorption of Cu^{2+} from aqueous solution using a mixture of tomato waste and corn stalk, which were oxidized with nitric acid. Heraldy et al. [39] studied the sorbent capacity for Pb^{2+} of tomato waste activated by sodium hydroxide, and the removal capacity obtained was 152 mg/g. The good lead removal properties and their improvement by the alkaline pre-treatment suggest that other procedures may also increase the sorbent qualities of tomato pomace and could be more convenient regarding cost, time consumption, green technology, ease of the procedure, etc.

The idea for the present study developed from the gap in the scientific literature concerning the use of tomato by-products as lead-adsorbing material and from some environmental issues: (1) the important annual wastewater volume; (2) the level of lead contamination in the water; and (3) the significant amount of tomato pomace generated by food industries. If additional studies shall confirm, in all respects, the opportunity to use tomato pomace for water decontamination, it will be possible to scale up the process to an industrial level.

As an innovative scientific strategy, the objective of the present study was to demonstrate that the pomace obtained after industrial tomato juice fabrication can be successfully used as an environmentally friendly alternative for lead removal from contaminated water. For this purpose, the tomato pomace was tested in two forms (the first one is eco-friendly, very cheap and easy to obtain, and the second one is a source of many other useful components): (i) dried (RT) and (ii) dried and extracted for the removal of water-soluble and lipo-soluble compounds (ET). The extraction of soluble compounds (most of them recognized for their important bioactive properties) to obtain ET was carried out considering their possible further valorization in food products (as natural antioxidants, coloring agents, fortifying agents, etc.), pharmaceutical products (supplements), cosmetics, etc. Assessments of the optimal pH conditions, mass studies, kinetics, and equilibrium isotherm tests were performed for both RT and ET. Also, the adsorption capacity (q) and the removal (%) of lead were calculated for all the experimental variants. Methods such as atomic absorption spectrometry, scanning electron microscopy, elemental chemical analysis with an energy dispersive X-ray detector and Fourier transform infrared spectroscopy were employed for the quantification of Pb and characterization of the newly obtained sorbents.

2. Materials and Methods

2.1. Reagents and Glassware Preparation

A lead-containing solution was used to simulate the Pb^{2+}-polluted water. A volume of 1000 mL stock solution containing 1000 ppm Pb^{2+} was prepared by dissolving 1.5985 g (Kern ADB 200-4, 210 g, 3 W, Balingen, Germany) of lead nitrate ($Pb(NO_3)_2$, Merck (Darmstadt, Germany), analytical grade, molar mass 331.2 g/mol) in distilled water (H_2Od). All further lead solutions were prepared by the dilution of the stock solution with distilled water.

The pH of the lead solutions was adjusted as necessary using diluted solutions of 0.1–1 N sodium hydroxide, NaOH (prepared from NaOH with purity $\geq$ 99.0%, Merck,

Darmstadt, Germany) and 0.1–1 N nitric acid, HNO_3 (analytical grade, 65%, Sigma Aldrich, Steinheim, Germany).

The laboratory glassware was kept for 24 h in 20% HNO_3 solution, and then it was thoroughly washed with distilled water to eliminate any possibility of lead contamination from previous tests that would reduce the accuracy of the results obtained.

2.2. Biosorbents Preparation

Tomato by-products were stabilized by drying at 60 °C until constant weight by using a convection laboratory oven (Biobase BOV-T30C, Jinan, China). Then, they were ground and sieved (1 mm, using IKA A 10 basic, 50 mL, 270 W, Staufen, Germany). The dried tomato pomace material obtained was named RT. As shown in Figure 1, RT was used to obtain the depleted tomato pomace (ET) by the removal of water-soluble and lipid-soluble compounds (which can be further valorized). The general purpose of the extraction was to earn free pores in the sorbent material, capable of retaining more of the lead ions. The protocol was similar to that described by Ungureanu et al. [32] with some improvements, as follows:

(1) To remove the water-soluble compounds, 15 g of dried RT sample was exactly weighed and mixed with 300 mL of distilled water. The sample was sonicated with an ultrasonic bath (HBM Ultrasonic Cleaner, GL 2.5 L, Moordrecht, The Netherland) at 50 ± 0.2 °C, for 20 min, at 40 KHz ultrasonic frequency. Next, it was placed for 30 min in a water bath (Biobase UC-40A, 10 L, 250 W, Jinan, China) with rotational stirring (100 rpm), at 85 ± 2 °C. The process was carried out twice, and then, the sample was filtered and the liquid was removed. The recovered solid fraction was dried in a convection laboratory oven at 45 °C.

(2) To remove the lipid-soluble compounds, the solid fraction obtained at (1) was mixed with 150 mL solvent mixture containing chloroform and methanol (1:2, *v/v*) and placed in an ultrasonic bath at 50 ± 0.2 °C for 20 min. After that, the sample was placed for 30 min in a water bath with rotary stirring (100 rpm) at 50 ± 0.2 °C and then the supernatant was discarded.

(3) The solid fraction obtained at (2) was washed with 100 mL ethanol (96%) and kept for 20 min on the water bath at 50 ± 0.2 °C, under stirring at 100 rpm. After decantation and removal of the supernatant, the process was repeated to remove all the lipophilic compounds.

(4) The solid fraction obtained at (3) was cleaned twice with 200 mL H_2Od and the third time was kept for 10 min under stirring (100 rpm) in the water bath at 50 ± 0.2 °C, then decanted.

(5) To improve the efficiency of extraction, ultrasonication was applied: 200 mL of distilled water was added to the solid part obtained at (4), placed for 20 min in the ultrasonic bath at 50 ± 0.2 °C, followed by decantation.

(6) The solid fraction collected at (5) was mixed with 200 mL H_2Od, stirred at 100 rpm for 20 min, at 50 ± 0.2 °C, in a water bath, and then decanted in order to remove, as much as possible, the small particles remaining attached in the pores of the obtained material.

(7) Finally, the solid fraction was dried using a convection laboratory oven at 45 °C, to a constant weight, and named extracted or depleted tomato pomace (ET). The used solvents were recovered by rotary evaporation and reused.

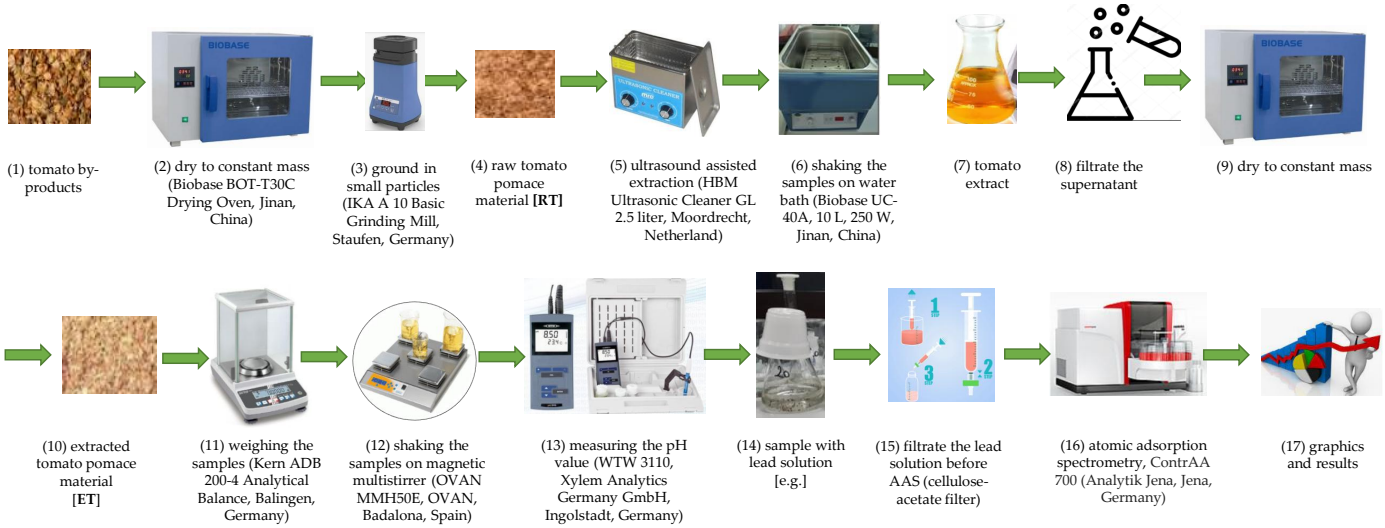

Figure 1. Biosorbents preparation from tomato by-products.

2.3. Characterization of Biosorbent Materials Derived from Tomato By-Products

2.3.1. Fourier Transform Infrared Spectroscopy (FTIR)

Fourier transform infrared (FTIR) spectra were recorded using an Interspec 200-X (Interspectrum, Tartumaa, Estonia) spectrometer at the instrumental resolution of 2 cm^{-1}. After drying and grinding the raw and extracted tomato pomace, the powder analyzing equipment was used to take the spectral measurements. This analysis was carried out in order to qualitatively describe the functional groups that were present on the surface of RT, RT–lead-loaded (RTPb), ET, and ET–lead-loaded (ETPb) [32,41].

2.3.2. Elemental Chemical Analysis (EDS) and Scanning Electron Microscopy (SEM)

Using an energy dispersive X-ray detector (EDS) (EDAX, AMETEK Inc., Berwyn, PA, USA) and a scanning electron microscope (SEM) with a Quanta 450 (FEI, Thermo Fisher Scientific, Hillsboro, OR, USA), the surface morphology of the biosorbent was examined both before and after lead adsorption. The TEAM V4.1 program was used to process the EDS spectra. The samples were taped on aluminum stubs using carbon double tape. Since the Quanta 450 operates in the environmental mode, samples did not require a conductive layer to be applied on their surface. With the samples positioned at 10 mm and at acceleration voltage of 15 kV, the measurements were performed in a high vacuum at 10^{-3} Pa pressure using an Everhart–Thorley detector. EDS measurements were performed immediately after micro-imagining of the samples [32].

2.4. Lead Analysis by Atomic Absorption Spectrometry

The lead concentrations of the initial and final (after lead-removal) solutions were assessed by atomic absorption spectrometry (AAS) with a ContrAA 700 spectrophotometer (Analytik Jena, Jena, Germany). In order to have the concentrations within the linear range of the instrument, dilutions were completed as necessary. The analysis was carried out using a 99.95% pure air/acetylene flame, performing triplicate readings at 217 nm. Calibration curves were produced prior to each analysis, and only the curves with a determination coefficient $R^2 > 0.995$ were accepted. The lead solutions were filtered before AAS analysis by 0.45 µm cellulose acetate filters [32].

2.5. Adsorption Studies

All the assessments were performed using lead solutions prepared as mentioned in 2.1, under rigorously controlled laboratory conditions, in order to ensure the accuracy of the

outcomes. The experimental data were mathematically modeled by non-linear regression adjustments (CurveExpert Professional software, 2.7.3 version) [32].

2.5.1. pH Study

In the adsorption process, the removal efficiency of heavy metals is significantly influenced by the pH. Therefore, in order to obtain the optimal pH value for maximum lead removal by tomato by-products, pH studies were performed at pH 2.5, 4.0 and 5.5. The main reason for selecting these pH values was that Pb-contaminated effluents typically have an acidic pH (between 2 and 4). Moreover, above pH 5.5–6.0, Pb^{2+} precipitation starts, with a maximum at pH 7.0–9.0 [32,42].

The pH test was performed with 1 g/L sorbent (RT or ET) and an initial Pb^{2+} solution of 20 ppm concentration. The contact time between the sorbent and lead solution was 240 min, at 120 rpm stirring (Biobase UC-A40, 10 L, 250 W, Jinan, China) and 23 ± 1 °C. During the experimental procedure, drops of HNO_3 or NaOH diluted solutions were used to maintain the pH at 2.5, 4.0 or 5.5, if there were registered variations over ±0.5. After 240 min contact time, the samples were filtered and the Pb^{2+} content of the final solution was analyzed by AAS. The mass balance formula (Equation (1)) was used to determine the amount of lead removed (q), expressed as mg Pb^{2+}/g sorbent material:

$$q = \frac{C_i - C_f}{m} V \quad (1)$$

C_i = initial lead concentration (mg/L), C_f = final lead concentration after adsorption (mg/L), m = mass of biosorbent (g), and V = volume of the solution (L).

Equation (2) was applied to determine the uptake efficiency (% of lead removal):

$$\%\ removal = \frac{C_i - C_f}{C_i} 100 \quad (2)$$

with the same significance of the abbreviations as in Equation (1).

2.5.2. Mass Concentration Assays

The amount of biosorbent plays an important role in adsorption studies. Therefore, to continue the investigation, the lowest dose of biosorbent with the best result in terms of lead adsorption must be established.

The mass study was performed using a biosorbent amount between 0.0125 and 0.5000 g in 50 mL solution (0.25–10.00 g/L), at the optimal pH value previously determined (4.0 ± 0.5) and for an initial solution of 20 ppm Pb^{2+}. The contact time of the RT or ET sorbent and the lead solution was 240 min, at a stirring speed of 120 rpm and a temperature of 23 ± 1 °C. For each sample, the adsorption capacity was calculated using Equation (1) and the removal efficiency of lead (%) was settled by Equation (2). The smallest sorbent concentration with the best results in terms of lead removal was denoted C_s (g/L) and employed in the following studies.

2.5.3. Biosorption Kinetic Studies

The outcomes of different contact times (5, 15, 30, 60, 120, 240, 360 and 480 min) at pH 4 ± 0.5, C_s of 0.5 g/L (value resulting from the tests carried out at 2.5.2), initial concentration of lead solution of 20 ppm Pb^{2+}, temperature 23 ± 1 °C and stirring speed 120 rpm were studied. Furthermore, the Lagergren model [43] (pseudo-first kinetic order and pseudo-second kinetic order) was plotted to experimental data by Equations (3) and (4), as follows:

$$q = q_e (1 - e^{-k_1 t}) \quad (3)$$

$$q = q_e \frac{k_2 q_e^2 \ t}{1 + k_2 q_e t} \quad (4)$$

where q = amount of lead removed for a contact time t, min (mg/g), q_e = biosorbed quantity at equilibrium (mg/g), k_1 = kinetic constant of pseudo-first kinetic order model (min^{-1}), and k_2 = kinetic constant of pseudo-second kinetic order model (g mg^{-1} min^{-1}).

For each contact time, the obtained results for RT and ET were compared using the independent-samples t test from IBM SPSS Statistics 21 software in order to assess the statistical significance (significance level 0.05).

2.5.4. Equilibrium Isotherms

Different initial lead concentrations ranging between 7.5 and 100 ppm were tested. The contact time was 240 min, and the rest of the experimental conditions applied were identical to those used in the kinetics tests.

The well-known equilibrium models Langmuir [44]—Equation (5) and Freundlich [45]—Equation (6) were plotted to the experimental data by non-linear regression:

$$q_e = \frac{K_L Q_{max} C_e}{1 + K_L C_e} \quad (5)$$

$$q_e = K_F C_e^{1/n_F} \quad (6)$$

where Q_{max} = the maximum biosorption capacity (mg/g), C_e = equilibrium lead concentration (mg/L), K_L = constant of Langmuir (mg/L), K_F = a constant of the adsorption system correlated to the removal capability (mg/g $(mg/L)^{-1/nF}$), and n_F = a constant expressing the acuity of adsorption. An isotherm was favorable only if n_F had super-unit values.

Although the Langmuir and Freundlich models are the most commonly used to describe isotherms, the particularity of the placement of the experimentally determined points required the application of a less used model, the linear isotherm model, expressed by Equation (7):

$$q_e = K_d \times C_e \quad (7)$$

where K_d represents the partition/adsorption coefficient.

For each initial Pb^{2+} concentration, the results obtained for RT, and respectively, ET were compared using the independent-samples t test from IBM SPSS Statistics 21 software in order to assess the statistical significance (significance level 0.05).

3. Results and Discussion

3.1. Biosorbents' Characteristics

3.1.1. Characterization by FTIR Spectroscopy

To identify the functional groups on the surface of the materials, FTIR spectroscopy was used. The complex structure of the surface of tomato pomace materials displays a wide range of functional groups, especially hydroxyl, carboxyl, carbonyl, ester, amine, etc.

Both biosorbents' spectra were acquired before (Figure 2a,b) and after their interaction with lead ions (Figure 2c,d). The main functional groups important for pollutant removal (e.g., the oxygen-containing functional groups around 3500 cm^{-1}) are shown in the following spectra (Figure 2).

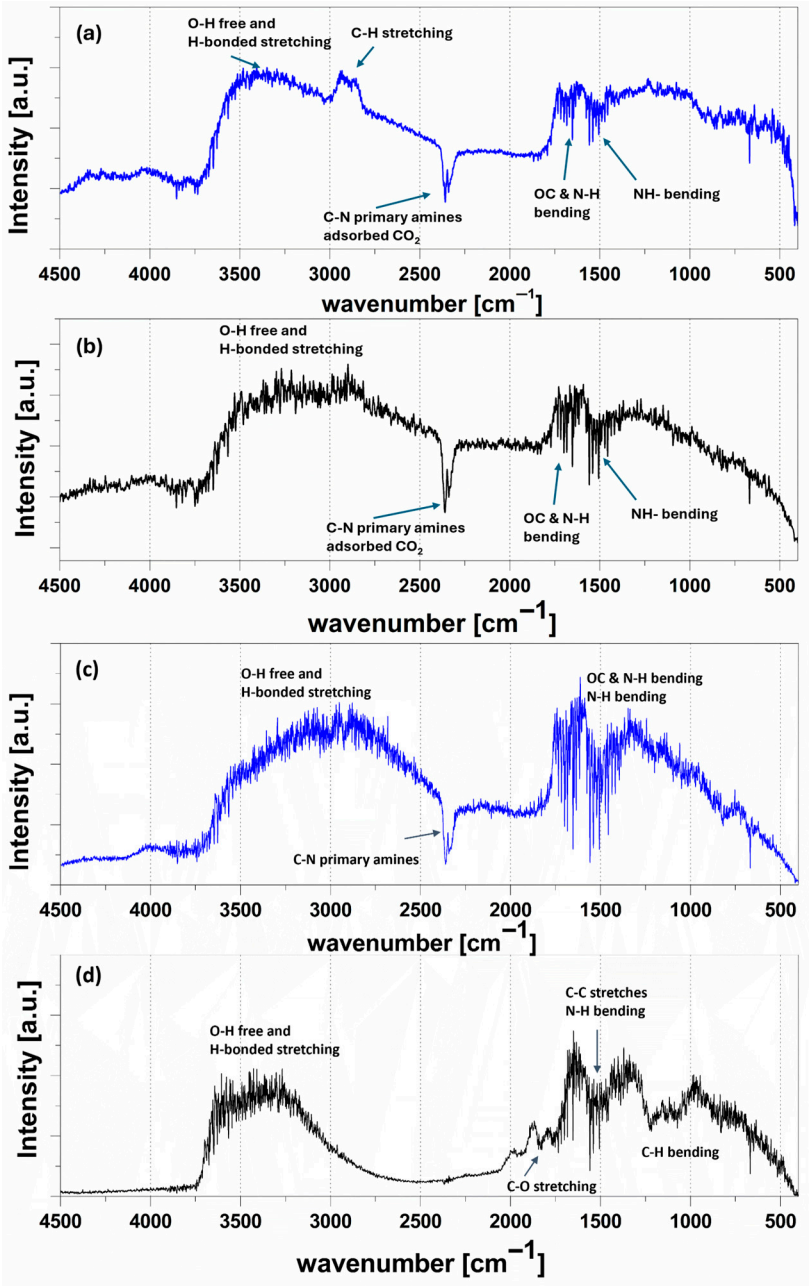

Figure 2. FTIR spectra of the biosorbents: (**a**) RT, (**b**) ET, (**c**) RTPb, and (**d**) ETPb.

FTIR data revealed a variety of chemical bonds for both materials, including C-H, C-C, C-N, N-H, O=C, and O-H bonds. Around 3500 cm^{-1}, there is a large band determined by O-H stretching from alcoholic and phenolic compounds, also observed by Heraldy et al., who studied a different type of biosorbent from tomato waste treated with NaOH [39]. Most probably, the mechanism of adsorption is represented by the formation of chelated compounds between lead ions and carboxylate and hydroxyl groups, as Pb^{2+} forms easily chelated compounds. All the studied biomaterials (before and after Pb-loading) revealed H-bonded stretching also related to the heavy metal adsorption. The C-H stretching decrease in RT after Pb-loading compared to the initial RT was due to lead adsorption. The findings concur with research published by Ungureanu et al. [32] in the case of a similar biosorbent derived from apple pomace.

No important shifts were observed in the characteristic peak positions following Pb adsorption. While minor changes in peak intensity or broadening were noted and discussed, substantial shifts in the wavenumber were not evident. This suggests that the

primary adsorption mechanisms might not involve strong covalent bond formation or significant alteration of the vibrational modes of the observed functional groups that would result in discernible shifts within the resolution of our FTIR analysis.

3.1.2. Characterization by SEM and EDS

The scanning electron microscopy presents the porous structure of the studied biomaterials (Figure 3). The porous aspect is more prominent and the cavities are larger and very clearly defined in the case of the ET sorbent, which is depleted in soluble compounds (Figure 3c). Contrarily, RT has a slightly disorganized appearance, with smaller and not so well-defined pores (Figure 3a), because it contains many water-soluble compounds (e.g., carbohydrates, organic acids, polyphenols, vitamins, etc.) and lipo-soluble compounds (e.g., lycopene, β-carotene, phytoene, phytofluene, tocopherols, phytosterols, etc.). After the lead adsorption on the material, in both cases, the porous appearance changes because of the lead retention and its inclusion inside the pores (Figure 3b,d).

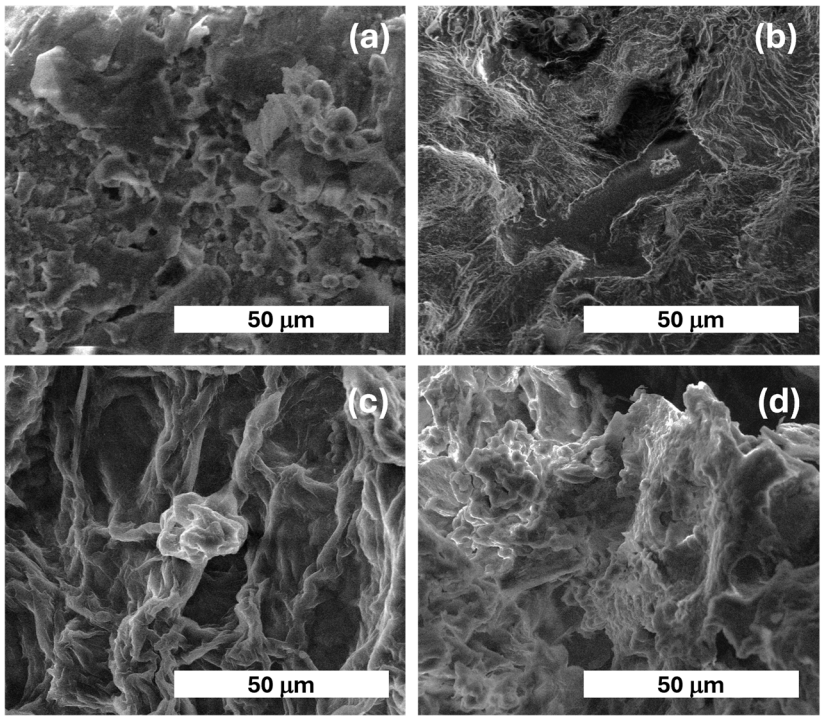

Figure 3. Microimaging of the materials' surface by SEM: (**a**) RT; (**b**) RTPb; (**c**) ET; and (**d**) ETPb.

Following extraction, some components leave the structure, thus creating "free places" where lead ions can bind. Therefore, the extraction steps improve the lead adsorption properties of the extracted material (ET). Similar results were obtained with apple pomace [32].

The results of the elemental chemical analysis (EDS) are semi-qualitative. Figure 4a–d show that in all the samples, carbon (43.95–54.69%), oxygen (25.89–41.48%) and nitrogen (12.74–24.27%) are the predominant chemical elements and the other elements are much less represented. As expected, comparing RT and ET, it can be noticed that after the extraction of water- and lipo-soluble compounds, some elements, such as P, Mg, S, Cl, and K, diminish or are not present anymore in ET. But the most important finding is the appearance of Pb in RTPb (0.04%) compared to RT, and in ETPb (0.90%) compared to ET, which confirms that both tomato-pomace-derived biomaterials are appropriate for lead removal from water. Moreover, the amount of lead removed from water is more important in the case of ET than in the case of RT, which proves that tomato pomace, after the extraction of soluble compounds, is a better sorbent than raw tomato pomace. However, an advanced study of these adsorbent materials, including techniques such as BET, XPS and XRD, would be

useful to validate these claims. For now, based on FTIR, SEM and EDS, it would be possible to propose only an inferred mechanism.

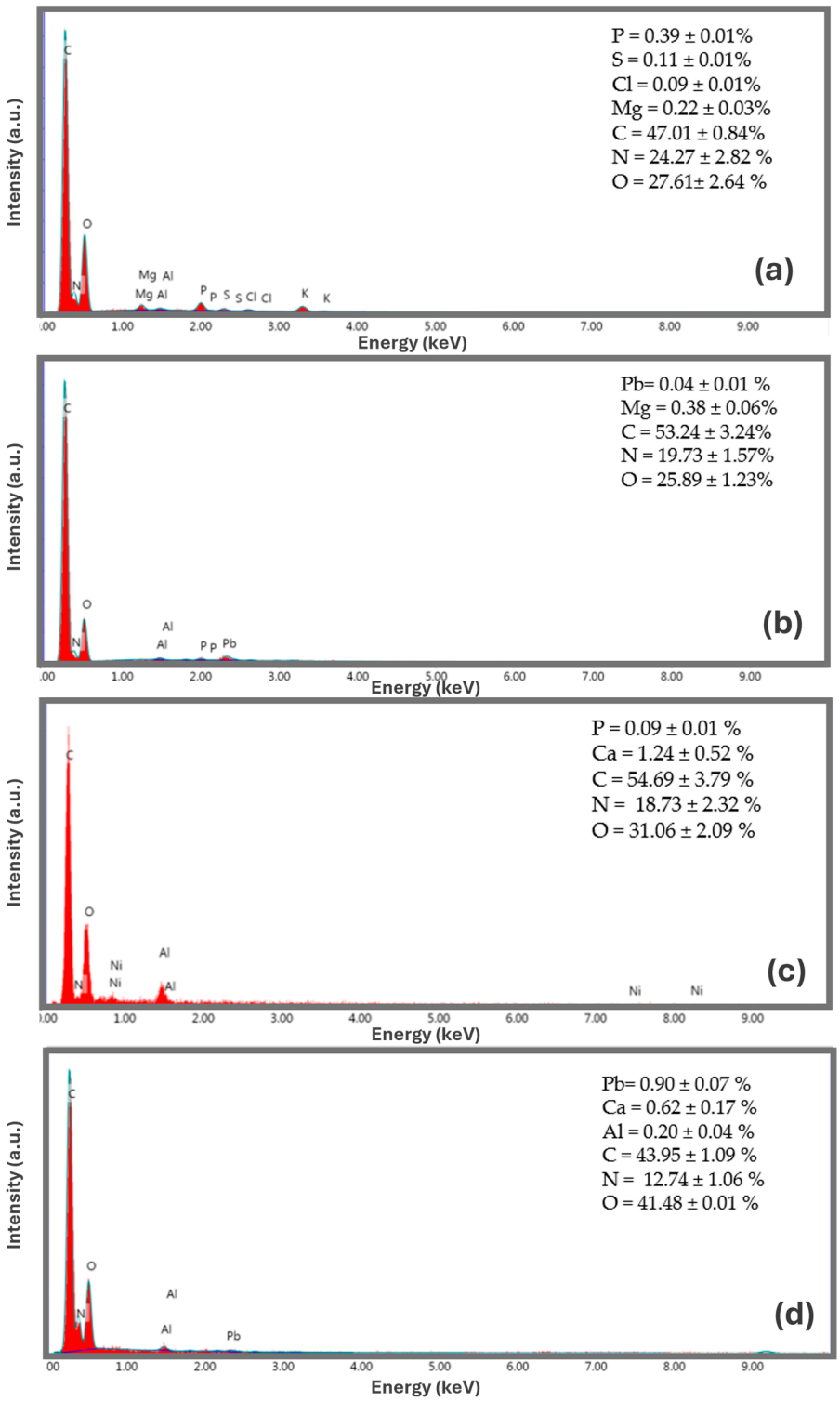

Figure 4. Elemental chemical analysis: (**a**) RT; (**b**) RTPb; (**c**) ET; and (**d**) ETPb.

The data obtained in this study are in accordance with similar studies of biomaterials similarly obtained from apple pomace [32]. There are three reasons for comparing the results of this study with those of Ungureanu et al. [32] on apple pomace materials: (1) the sorbent materials are prepared using similar techniques, (2) both apple and tomato pomaces

exist in large (and comparable) quantities, and (3) to enlarge the variety of possible-to–use sorbents for lead removal from water.

3.2. Influence of pH on the Lead Removal

Regardless of the nature of the adsorbent or pollutant, the pH is a critical parameter for the adsorption process. In this context, previous studies suggest that lead adsorption is also largely dependent on the pH, with acidic conditions being of primary interest because they maximize the lead retention [32,46]. Furthermore, lead-contaminated wastewater, which originates from many industries (such as mining, metallurgy, etc.), usually has an acidic pH, and this reinforces the practical reason why the study was conducted at pHs of 2.5, 4.0, and 5.5. According to lead speciation, at pH $\leq$ 5.5, the free Pb^{2+} ion, which is soluble and accessible, is the most common species. At higher pH (6.0–10.0), Pb^{2+} is precipitated in solution, in the form of insoluble hydroxide $Pb(OH)_2$. Above pH 10.0–11.00, lead becomes soluble again, forming anionic complexes $[Pb(OH)_3]^-$ and $[Pb(OH)_4]^{2-}$ [47,48].

In the present study, both tested sorbents provided the best lead adsorption and removal capacity at pH 4.0. Thus, according to Figure 5, for RT, the lead retention capacity at pH 4.0 was 89.83%, while for the ET sample, it reached up to 99.22%. The amount of lead removed by RT was 15.20 mg Pb^{2+}/g biomaterial and by ET was 22.11 mg Pb^{2+}/g biosorbent.

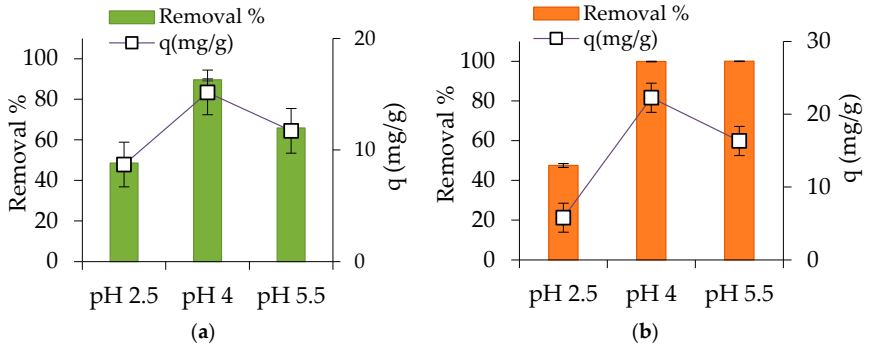

Figure 5. pH effect on lead adsorption and removal capacity, C_0 = 20 ppm Pb^{2+}, T = 23 ± 1 °C, 4 h contact time, C_s = 1 g/L: (**a**) RT and (**b**) ET.

Therefore, the optimal pH value for the present study was considered to be 4.0, and the rest of the experiments were performed at pH 4.0 ± 0.5. Since this pH is not within the range of 5.5–10.0, lead precipitation should not occur, and consequently, no experimental control (e.g., blank without adsorbent) was included to distinguish the effects of adsorption from those of precipitation.

Heraldy et al. [39] also find that pH 4.0 is the most appropriate for Pb^{2+} removal from water by tomato waste activated by treatment with sodium hydroxide, but they obtained less than 97% removal, compared to more than 99% in case of ET [39].

3.3. Influence of Adsorbent Dosage

The dose of adsorbent is essential for the best removal of a heavy metal at a specific initial metal concentration. Figure 6 displays the experimental data regarding the influence of the adsorbent amount on the removal capacity of RT and ET. The retention percent increased gradually from 0.25 to 1.00 g/L for both RT and ET, and from 1.00 to 10.00 g/L, the growth was much diminished. The increase can be explained by the augmentation of the number of sorbent active sites, which capture a larger amount of Pb^{2+}, and the subsequent decrease is due to a loss of efficiency in the use of the adsorbent surface because of the overlapping of the active sites of the material. If this increase in removal efficiency is superimposed on the adsorption capacities, a pronounced decrease in this capacity is observed with the increase in the adsorbent amount, precisely due to the excessive growth

of the positions available for fixing lead ions, which are mutually blocking and become an impediment to lead retention on the active sites of the biosorbent.

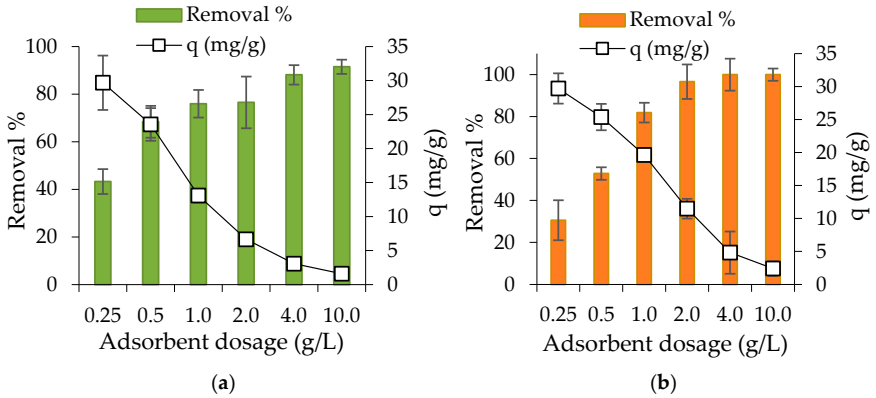

Figure 6. Removal effectiveness and adsorption capacity of (**a**) RT and (**b**) ET at various biosorbent concentrations (0.25–10.00 g/L), C_0 = 20 ppm Pb^{2+}, pH 4 ± 0.5, T = 23 ± 1 °C, 4 h contact time.

In the case of the RT sample, the maximum lead removal was 91.5% for 10 g/L biosorbent amount, but the highest experimental adsorption capacity was 25.96 mg/g at 0.5 g/L adsorbent material. For the extracted material (ET), the maximum value for lead removal efficiency was 100% for 10 g/L biosorbent amount, but the experimental adsorption capacity was 25.38 mg/g at 0.5 g/L adsorbent concentration. Although a biosorbent concentration of 0.25 g/L apparently shows the best adsorption capacity, it was not selected for further tests due to the possibility of the error being high due to the very small amount of adsorbent. In conclusion, the optimal adsorbent dose was considered to be C_s = 0.5 g/L, and it was used for the following kinetics and equilibrium studies.

The optimal dose of biosorbent was chosen as the minimum quantity that could be used so as to avoid a possible error due to weighing a too small quantity, but small enough to prove the adsorption efficiency of the used material.

Permatasari et al. [49] found that, in case of NaOH-activated tomato waste, the best removal of Pb^{2+} was achieved for 0.1 g sorbent/25 mL (4 g/L), which is four times more concentrated than in the current study.

3.4. Biosorption Kinetics

The contact time between the sorbent and the heavy metal solution is another essential parameter for an adsorption study. Figure 7 illustrates the influence of the contact time (between 5 and 480 min) on the adsorption capacity of both studied biosorbents.

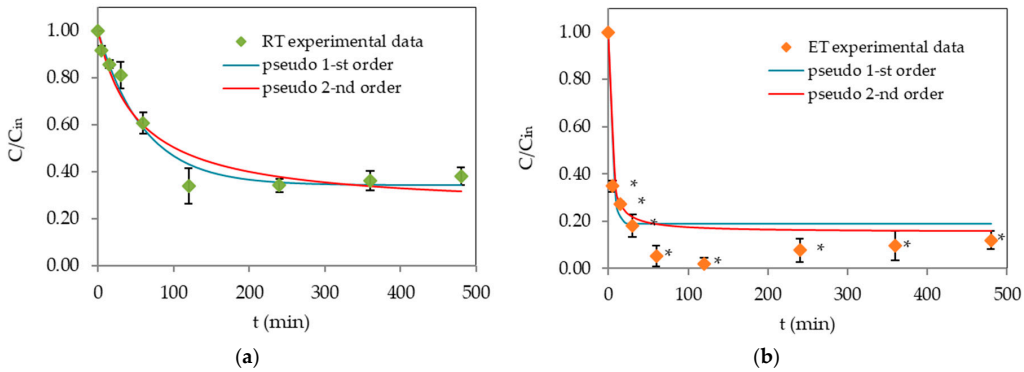

Figure 7. Influence of contact time on Pb^{2+} biosorption by (**a**) RT and (**b**) ET at C_0 = 20 ppm Pb^{2+}, C_s = 0.5 g/L biosorbent, T = 23 ± 1 °C (experimental data and pseudo-1st and pseudo-2nd order model); "*" for ET data in (**b**) indicates the values that are significantly different from those for RT data in (**a**) for the same contact time.

The obtained results show that, in the case of RT, the system adsorbent–adsorbate requires about 120 min to reach equilibrium, while for ET the process is faster, and in less than 60 min, the reaction reaches the equilibrium point. Comparing for each contact time, with the results obtained for RT and ET, it can be noticed that they are statistically different, excepting the initial moment (t = 0).

At 120 min of contact between lead-contaminated water and tomato by-products, RT shows an experimental adsorption capacity of 25.07 mg/g, and at 60 min, ET 32.9 mg/g.

Similarly, in the case of sodium hydroxide-activated tomato waste, Permatasari et al. [49] found that 75 min is the equilibrium contact time for the maximum percent of lead removal.

The pseudo-first order (Table 1) and pseudo-second order (Table 2) models predict the values of the adsorption capacities at equilibrium, q_e, close to the experimental data for both biosorbents. However, the best fitting is for the pseudo-first order model, when R^2 is 0.97 for RT and 0.95 for ET (Table 1).

Table 1. Pseudo-1st order model of kinetic study adjustments (value ± standard error).

Sample	k_1 (min^{-1})	q_e (mg/g)	R^2
RT	0.017 ± 0.002	24.94 ± 1.13	0.97
ET	0.23 ± 0.05	30.84 ± 0.98	0.95

Table 2. Pseudo-2nd order model of kinetic study adjustments (value ± standard error).

Sample	k_2 (g mg^{-1} min^{-1})	q_e (mg/g)	R^2
RT	0.0007 ± 0.0003	28.88 ± 2.61	0.96
ET	0.012 ± 0.003	32.07 ± 0.84	0.81

Kinetic analysis shows the better fit of the pseudo-first order model, as the R^2 coefficient is higher than the pseudo-second order model. This result does not align with the results obtained in most biosorption studies, which indicate the pseudo-second order model to be the most appropriate. However, there are studies that report that the pseudo-first order model provides a better fit. For example, Ungureanu et al. [50] performed a biosorption study on spruce bark, and the pseudo-first order model also provided a better fitting for the same initial lead concentration (20 ppm). A possible explanation could be that biosorbents are obtained from biological material and the structure of the raw material is complex and extremely heterogeneous. Some biosorbents have a homogeneous surface, with the active centers having the same affinity for the metal ion, while for other biosorbents, the metal binds to two active centers.

In addition, monolayer adsorption and the existence of accessible sites for biosorption are assumed by the Langmuir model. Only one adsorbate entity can be fixed by each active site. All the active sites have the same adsorption energy, which is independent of the presence of nearby adsorbed species [31,43,44,48].

Furthermore, the exponential distribution of active sites and their energies, as well as the surface heterogeneity, is empirically expressed by the Freundlich adsorption isotherm as follows: $n_F > 1$, favorable isotherm; $n_F \leq 1$, unfavorable isotherm [31,43,44,48].

Regarding biosorption, four mechanisms can generally be identified: chemisorption, physisorption, microprecipitation and oxidation/reduction, which can occur separately or together [51]. Compared to other previous research [33,52], in this study, chemisorption could be the main mechanism that could explain the biosorption of lead ions on tomato pomace, most likely through the attraction/chelation between the positively charged Pb^{2+} ions and the negatively charged (-COO^-)/(OH^-) groups revealed by FTIR analysis.

3.5. Equilibrium Studies

Information about the maximum adsorption capacity of a biosorbent, about its surface characteristics, and about the affinity between an adsorbate and biosorbent is provided by studying the equilibrium isotherm.

The results presented in Figure 8a,b show that, even for very low initial lead concentrations (7 ppm), both RT and ET present removal capacities. The experimental data prove, for all the initial Pb^{2+} concentrations, better lead removal capacities for ET than for RT, results which are statistically supported (Figure 8).

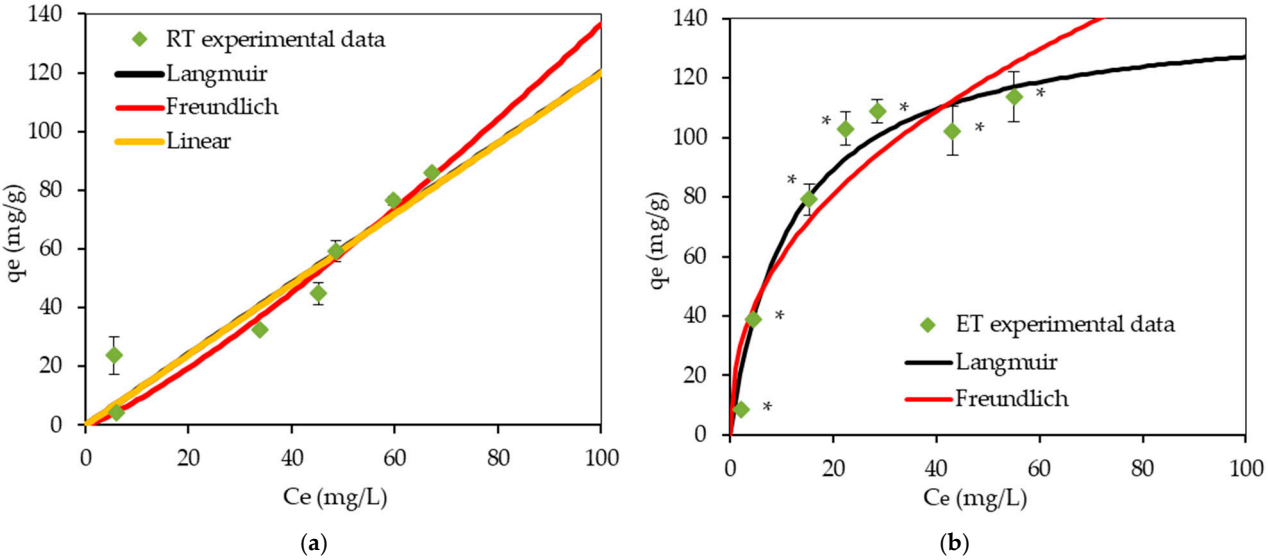

Figure 8. Isotherms (experimental data and model curves) for Pb^{2+} biosorption on (**a**) RT and (**b**) ET at *Cs* = 0.5 g/L biosorbent, pH = 4 ± 0.5, T = 23 ± 1 °C, 4 h contact time and different initial lead concentrations; "*" for ET data in (**b**) indicates the values that are significantly different from those for RT data in (**a**) for the same initial Pb^{2+} concentration.

The well-known equilibrium models Langmuir [44] and Freundlich [45] were fitted to experimental points by non-linear regression. But Figure 8 shows that the amount of adsorbed lead increases practically linearly with the equilibrium concentration (C_e). Consequently, a linear isotherm model was also chosen to describe the equilibrium isotherm by Equation (7).

The data provided by the Langmuir, Freundlich, and Linear models are presented in Table 3, Table 4 and Table 5 respectively.

Table 3. Langmuir model of equilibrium study adjustments (value ± standard error).

Sample	Q_{max} (mg/g)	K_L (L/mg)	R^2
RT	90.91 ± 2.84	0.03 ± 0.03	0.49
ET	142.18 ± 13.22	0.08 ± 0.02	0.97

Table 4. Freundlich model of equilibrium study adjustments (value ± standard error).

Sample	$1/n_F$	K_F (mg g^{-1} (L $mg^{-1})^{1/n}$)	R^2
RT	1.21 ± 0.21	0.52 ± 0.65	0.91
ET	0.43 ± 0.53	22.29 ± 7.61	0.91

Table 5. Linear model of equilibrium study adjustments (value ± standard error).

Sample	K_d (L g^{-1})	R^2
RT	1.2001 ± 0.0481	0.97

As it is easy to observe in Table 4, the Langmuir model applied for the RT isotherm is inadequate due to a low coefficient of determination (R^2 = 0.49) compared to the Freundlich model, which better adapts to experimental points, having an R^2 of 0.91. But the best representation is realized by the linear model, which has a coefficient of determination R^2 of 0.97.

For the ET biosorbent, the situation is different. Better adaptation to the experimental data is seen for the Langmuir model, with R^2 = 0.97.

The adsorption of lead ions (Pb^{2+}) onto tomato pomace is a process dependent on the complex structure of the biosorbent. By analyzing the structural changes before and after metal loading, we can identify how this "waste" material captures heavy metals such as lead. The FTIR spectra highlighted a surface rich in oxygen-containing functional groups. Specifically, the broad band at 3500 cm^{-1} signifies O-H stretching from phenolic and alcoholic compounds. These groups, alongside carboxylate functions, serve as active sites. The shift or decrease in C-H stretching intensity after loading directly confirms that the lead ions are not just sitting on the surface but are actively interacting with the biomass structure. Lead ions have a high affinity for oxygen-rich ligands; they effectively "clamp" onto the hydroxyl and carboxylate groups. More than that, the fact that the adsorption kinetics are best described by the pseudo-first order model, with a higher R^2 coefficient compared to the pseudo-second order model, indicates a rapid initial uptake, when lead ions rapidly occupy the accessible phenolic and carboxylic sites of the tomato pomace surface.

The current study proposes a new approach for tomato pomace valorization in a sustainable, environmentally friendly, and accessible way, which supports the circular economy. The used by-product is considered a food waste obtained after tomato processing, with large availability worldwide, in a high quantity, at low or no price (being a waste that industry wants to get rid of). Therefore, this research involves a simple, clean, and efficient method to reduce the tomato waste amount. A strength of this research is that previous similar studies also reported lead biosorption from water using agri-food by-products, such as peels of lemon, orange, bean, artichoke, coffee, spent coffee, grape marc, apple pomace, etc. (Table 6).

Table 6. Lead adsorption capacity of different biosorbents.

Biosorbent	Adsorption Capacity * (mg/g)	Initial Lead Concentration (mg/L)	pH	Sorbent Dose (g/L)	Stirring Speed (rpm)	Reference
Lemon peels	20.62	20	3.0	0.5	160	[25]
Bean peels	51.81	20	3.0	0.5	160	[25]
Artichoke peels	69.93	20	3.0	0.5	160	[25]
Orange peels (fresh/dried/powder)	37.20–56.70	100	5.0	0.1	n.m.	[26]
Lemon peels (fresh/dried/powder)	58.00–72.50	100	5.0	0.1	n.m.	[26]
Coffee (shell/pulp)	4.80–54.05	n.m.	4.5–5.0	n.m.	n.m.	[29]
Coffee waste, biorefined (citric acid/pyrolysis)	54.05–159.50	n.m.	3.5–5.0	n.m.	n.m.	[29]

Table 6. *Cont.*

Biosorbent	Adsorption Capacity * (mg/g)	Initial Lead Concentration (mg/L)	pH	Sorbent Dose (g/L)	Stirring Speed (rpm)	Reference
Spent coffee beans	87.02	n.m.	3.0–4.0	n.m.	n.m.	[29]
Raw coffee beans	22.90	n.m.	5.0	n.m.	n.m.	[29]
Coffee pulp	24.10	100	2.0	0.5	100	[53]
Apple pomace, raw	44.6	20	5.5	0.5	120	[32]
Apple pomace, extracted	48.6	20	5.5	0.5	120	[32]
Merlot grape pomace, extracted	40.1	20	5.5	0.5	120	[33]
Sauvignon Blanc grape pomace, extracted	63.8	20	5.5	0.5	120	[33]
Tomato pomace, raw	90.91	20	4	0.5	120	Present study
Tomato pomace, extracted	142.18	20	4	0.5	120	Present study

n.m. = not mentioned; * = maximum adsorption capacity predicted by Langmuir model.

Many factors may influence the lead adsorption capacity of a material, from the specie and variety of the original plant to the technological conditions of processing in order to obtain the by-product, the procedure used for the stabilization/extraction/treatment of sorbent material, the particle size, and the methods applied during the assays that test the sorbent (initial concentration of the metal in the polluted water, presence of interfering compounds in the water, pH, temperature, dose of sorbent, stirring speed, etc.). Therefore, the same type of sorbent may have very different adsorption capacities. For example, Ergüvenerler [25] found for lemon peels a lead adsorption capacity of 20.62 mg/g, while in different conditions, Husoon [26] obtained for fresh, dried, and powder lemon peels 58.00–72.50 mg/g. Furthermore, compared to fresh and dried pieces, the powdered form of lemon peel adsorbed more Pb^{2+}. In the same study, Husoon observed a smaller adsorption capacity of orange peels (fresh, dried, or powder), 37.20–56.70 mg/g, compared to lemon peels (Table 6).

A highly studied sorbent is the one based on various forms of coffee [29,30,53]. Skorupa [29] found that spent coffee beans (87.02 mg/g) adsorb lead better than raw coffee beans (22.90 mg/g), but the coffee waste biorefined can adsorb much better, up to 159.50 mg/g (Table 6). Also, Seniūnaitė et al. [30] studied the removal of lead from aqueous solutions using coffee grounds at various quantities of biosorbent (0.5, 1.0, 1.5, and 3.0 mg/L) and different particle sizes. Their results confirm that both used fractions (particle size > 200 µm and, respectively, <200 µm) are efficient.

Comparing the maximum adsorption capacity (predicted by the Langmuir model) of tomato pomace from the present research with apple and grape pomaces prepared similarly, and previously studied (Table 6), the following can be noticed:

- Extracted pomaces are better lead adsorbents than raw pomaces, and this can be explained by the fact that, during extraction, the soluble components leave from the material's structure, and their place can be taken by lead ions.
- Sauvignon Blanc grape pomace is a better sorbent than Merlot grape pomace.
- Tomato pomaces (raw and extracted) are better lead adsorbents than apple and grape pomaces when analyzed in similar conditions.

Consequently, the present study is in accordance with modern research directions concerning environmental depollution and agri-food waste valorization within the circular

economy, and dried tomato pomace (in raw or extracted form) could be successfully used as a lead-decontaminating biomaterial (even at industrial scale). More than that, the liquid fractions resulting during ET preparation (from the extraction of soluble compounds from the tomato pomace) can be further valorized as sources of natural bioactive compounds (pigments, antioxidants, etc.) for obtaining value-added products (e.g., jelly candies, etc.), similar to those obtained from apple pomace extract [54]. The use of organic solvents is a weak point from the perspective of green chemistry, but this trade-off is necessary for the recovery of lipo-soluble compounds from tomato pomace.

4. Conclusions

The present research studied tomato pomace's efficiency for lead removal from a synthetic system. It proves that tomato pomace can be successfully used for lead removal from water in both studied forms: RT—the simple one (dried only), which is eco-friendly, very cheap, and easy to obtain, and ET—the one depleted in soluble compounds. Both forms are very efficient in terms of lead removal from water, but better results were obtained for the extracted pomace, which also has another important advantage, as the extracts resulting from ET preparation can be reintroduced into the food industry (as colorants, antioxidants, fortifying agents, etc.), pharmaceutical products, or cosmetics, sustaining the circular economy. As future research, the current findings require validation for real systems applications. The present study could be completed with similar tests for other heavy metals' removal and with assays on real polluted wastewater of different origins. If the tomato pomace will be applied at industrial scale for water decontamination, it will induce environmental, economic, and health advantages: environmental by removing the heavy metal and by reusing a waste (the tomato pomace), according to the "zero waste" principle; economic by formulating a cheaper sorbent than other materials; and health advantage by providing natural colorants/antioxidants/fortifying agents for food products.

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Abbreviations

The following abbreviations are used in this manuscript:

RT	Raw tomato pomace
ET	Extracted tomato pomace
RTPb	Raw tomato pomace loaded with lead
ETPb	Extracted tomato pomace loaded with lead
AAS	Atomic absorption spectrometry
EDS	Elemental chemical analysis
FTIR	Fourier transform infrared spectroscopy
SEM	Scanning electron microscopy/scanning electron microscope

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