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Multiproxy Approach to Reconstruct the Fire History of *Araucaria araucana* Forests in the Nahuelbuta Coastal Range, Chile

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Abstract: Multiproxy reconstructions of fire regimes in forest ecosystems can provide a clearer understanding of past fire activity and circumvent some limitations of single proxy reconstructions. While inferring fire history from scars in trees is the most precise method to reconstruct temporal fire patterns, this method is limited in *Araucaria araucana* forests by rot after fire injuries, successive fires that destroy the evidence and the prohibition of sample extraction from living Araucaria trees. In this context, dendrochemical studies in Araucaria trees and charcoal analysis from sediment cores can complement and extend the time perspective of the fire history in the relictual Araucaria-Nothofagus forests of the coastal range. We used dendrochemical, fire scar and charcoal records from the Nahuelbuta Coastal Range (37.8° S; 73° W) spanning the last 1000 years to reconstruct the fire history. The results indicate that periods with higher fire activity occurred between 1400 and 1650 AD. Long-term changes in the fire regime are related to increased climate variability over the last 1000 years, and especially with the arrival of settlers to the area after 1860 CE. The most severe fire events in the Nothofagus and Araucaria forests occurred when suitable fire-prone conditions were superimposed with high human densities.

Keywords: dendrochemistry; *Araucaria araucana*; Nahuelbuta range; fire regimes; ICP-MS

1. Introduction

Fire history is critical for understanding the potential effects of changing climate and human land use on the forest landscape [1–4]. Under the current climate change scenario, south-central Chile (37°–42° S), characterized by sharp declines in rainfall and increases in the recurrence of drought and heat waves [5–8], is expected to see an increase in the recurrence and magnitude of forest fires [9–16]. Fire occurrence in this area has also been linked to the climatic effects of Southern Annular Mode (SAM) [17,18] as well as to El Niño Southern Oscillation (ENSO) variability [17,19,20].

Fire is a major disturbance process shaping the Araucaria forest landscape in the Andean and Coastal ranges of north-west Patagonia [18,21–23]. *Araucaria-Nothofagus* forest ecosystems have evolved with fire and some plant species, such as *Araucaria araucana* (Molina) K. Koch (Araucaria), have developed distinct strategies to cope with this process [24–27]. The fire regime of Araucaria forests is characterized as mixed severity, where fires may leave behind unburned patches along with patches burned with a low, moderate and high severity (>75% overstory tree kill) [28]. Low-severity surface fires can occur at relatively short intervals and are generally associated with more open sites dominated by Araucaria mixed with *Nothofagus antarctica* (G. Forster) Oerst. woodlands. Stand-replacing crown fires are common in more productive mesic mixed species stands of *Araucaria–Nothofagus* forests where emergent, thick-barked Araucaria trees frequently survive [29].

Over recent decades, *Araucaria-Nothofagus* forests have been affected by large high-severity fires, both in protected areas (national parks and reserves) and on private land [18,21,27,30,31]. Although an increase in the recurrence of fires in Andean Araucaria forests has been documented since the Euro-Chilean period (1883–1970) [18,21], fire events occurring in the last 50 years have been considered exceptional in terms of their frequency, area burned and fire severity [28,30].

Araucaria araucana is a tree species of recognized cultural, scientific and socio-economic importance. This long-lived conifer is endemic to the temperate forests of Chile and Argentina [24,25,32,33], and classified as Vulnerable (VU) in its Andean distribution and Endangered (EN) in its relic distribution in the Nahuelbuta Coastal Range [34–36]. It is estimated that the original area of Araucaria forests at the arrival of European colonizers was about 500,000 hectares, but this number was reduced by half by the end of the 20th century, with only 254,000 hectares remaining [37]. The effects of the European-Chilean colonization of southern Chile, and subsequent logging in the Araucanía region in the 20th century, triggered a drastic reduction in forests, leaving those remaining in a critical state [33]. Despite being protected by Chilean law (Decreto N° 43 del Ministerio de Agricultura 1990, which prohibits the cutting and logging of these forests), they are currently at a high risk of degradation, either by illegal logging [19], cattle grazing [26,27,38], the invasion of exotic species [39,40], seed predation by non-native species and the intensive collection of edible seeds [25], or fire-induced disturbances [30,41].

Several studies regarding the fire regime of *A. araucana* forests have been conducted in the Andean part of its distribution [18,21,28,29], but little is known about the historical range of fire variability in the populations growing in the Coastal Mountain Range, especially in the Nahuelbuta Range. This is of special interest, since this area has a different history of occupation than the Andes, in addition to not having volcanic activity, which can be linked in some degree to the fire activity in the Andes. Knowing the fire regime in Araucaria forests in different areas of its distribution, especially in the Nahuelbuta Coastal Range, would allow us to assess the different degrees of disturbance and fire dynamics in these forests to inform management and restoration plans for these unique ecosystems in protected and private areas.

Fire history has been reconstructed through various environmental proxies, such as fire scars on fire-affected trees, stand age classes, increases or decreases in tree growth ring width, macroscopic charcoal particles and pollen and spores in peatbog sediments, among others [42–45].

The study of fire scars is based on the analysis of the growth rings of trees in segments of the trunk where the fire burned through the bark causing the death of the cambium and a subsequent scarring of the damaged area [46,47]. These scars make it possible to date fires with an annual or in some cases seasonal resolution throughout the life of the tree [46,47]. Often after fire, an abrupt radial growth response (release) occurs associated with the healing callus [47]. However, due to the low presence of old trees (>500 years), few old fire records can be dated. In addition, fire sensitivity differs amongst trees and tree species, so not all store records of fire in their wood anatomy, and a loss of records after successive large fires over time has been documented, precluding a complete fire reconstruction [21,46,48].

Fossil charcoal, on the other hand, is the most frequently used technique to reconstruct fire history from lake deposits, as it derives from the incomplete combustion of plant tissue [49]. Charcoal dating in lake sediments has been used to date fires in Chile up to 26,000 years old [50]. It can cover larger burned areas than fire scar records, especially because the macroscopic charcoal particles from fires inside a catchment area are deposited just as sediment from that area is deposited in fluvial and lacustrine environments [51]. Thus, this type of record catalogues fire events on larger spatial and temporal scales that are not necessarily close to a given study site (patch scale). The temporal resolution depends on the sedimentation rates of each lake or swamp; however, compared to the ring chronology yearly resolution, it is only possible to date fire events at a decadal to centennial to millennial time resolution [51].

These paleoenvironmental proxies are by definition an approximation of reality and are affected by multiple processes [44]; hence, it is important to obtain multiproxy information to enrich and complement these records and to develop a better understanding of the fire regimes over time.

Trees have been shown to chemically respond to variations in the environment by retaining chemical elements in their growth rings [52], the concentrations of which can be indicators of environmental change over time [53,54]. Dendrochemical studies have been successfully utilized to study trace metal deposition in tree rings and have been widely used to monitor environmental changes in soil and atmosphere [55]. Alternative dendrochemical techniques have been used to study the effects of atmospheric pollution [56–58] and volcanic eruptions [59–62]. Wildfires generate large numbers of pollutants, including trace elements [63,64]. During fire events, several elements (macro and trace nutrients) are released, deposited in the soil [63,64] and then absorbed and stored by trees in their woody tissues [65]. In effect, some studies show a variation in the chemical composition of wood surrounding fire scar samples [60,66,67]. Moreover, changes in the concentration of N and P in tree rings before and after fire events have been described [68], and an increase in the concentrations of various elements has been observed in tree rings for up to nine years after a fire [60]. Increases of B, Al, Mn and Fe have also been found to be useful as indicators of fire occurrence, further corroborating the potential of dendrochemistry for studying fire history [69]. However, the mechanisms by which the elements are fixed inside tree rings are not yet fully understood and show mismatching results [54], making dendrochemistry one of the most challenging lines of dendrochronology at the present time. One way to confirm dendrochemical results is to calibrate them with other proxy records, which is especially needed to assess the potential of dendrochemical results to detect past fires and other disturbance regimes.

The dendrochemical potential of *A. araucana* has been recently explored to date volcanic eruptions in the Andes Mountains, showing promising results that indicate the species' ability to record pulses of chemical elements released in eruptions of varying explosivity [70,71]. Ref. [72] point out that evergreen coniferous species may be more sensitive to chemical changes in the environment than deciduous species. Araucaria, being a long-lived evergreen conifer with individuals exceeding 1000 years of age [32], can potentially provide long dendrochemical records of past environmental conditions. Our hypothesis in this work is that the smoke and particles emitted in forest fires could alter *A. araucana's* uptake of chemical elements through its leaves or roots and be reflected in the concentrations of these elements in its growth rings through time. This approach could complement the information provided by other environmental proxies used to study fire dynamics, such as fire scars and sedimentary micro-carbon. Furthermore, unlike fire scar sampling, dendrochemistry uses a less invasive technique for sample acquisition; therefore, it can be a very useful tool in those places where scar samples cannot be taken (e.g., national parks).

In this context, this work is the first to evaluate the use of dendrochemical records to study fires in *Araucaria araucana* forests by comparing them with records from fire scars on trees and charcoal records in sediment cores. This information may be key to achieving high-resolution fire records in Araucaria forests for the last millennium, helping to clarify and better interpret both the impact of the arrival of Spaniard conquistadors on the fire regime in the Araucanía region and to identify the periods of high climatic variability and the influence of high temperatures on the Araucaria forest landscape.

2. Materials and Methods

2.1. Study Area

Our study area was the Nahuelbuta Coastal Range (37°–38°50′ S, Figure 1), located between the Bío-Bío and Imperial rivers, with an extension of 175 km in length [73] and a maximum elevation of 1525 m a.s.l. (Figure 1A). The Nahuelbuta Coastal Range is a biogeographically relevant place since it remained free of periglacial processes during the glacial events of the Quaternary, developing a more stable biogeographic history compared to the Andes [73]. This allowed a rich diversity of narrow-range endemic flora and fauna to concentrate in this mountain range, with some vascular plants whose only distribution is in this mountain range [73]. Moreover, it represents the predominant distribution of the Araucaria forest on the coast. This mountain range is characterized by a maritime climate with a Mediterranean influence, reflected in a distribution of maximum rainfall in winter and relatively dry and hot conditions during the summer months (December–March) [35,74]. The amounts of annual precipitation vary between 1500 and 3000 mm, influenced by snow at high altitudes, while the average annual temperature values have a wide variability, with minimum temperatures in July and maximum temperatures in February [38]. In general, soils derive from metamorphic material and in some sectors from granite [38,75]. Unlike the Andes, this mountain range has no volcanic influence, which is why the fire signals can only be due to forest fires, allowing us to remove some of the noise in the records.

During prehistoric times, the valleys of the Nahuelbuta Coastal Range presented different socioeconomic strategies of land use, from the nomadic hunter-gatherers of the early and middle Holocene to the complex agro-pottery systems of the late Holocene (1000–1500 CE), with the presence of ceremonial mounds (kuel) in valleys such as Purén and Lumaco [76]. This gradual sociocultural complexification of lowland areas parallels the continuous use of high-altitude ecosystems and their Araucaria forests, which for the Mapuche people ancestrally correspond to a culturally and ritually significant space in which the gathering of species such as Araucaria fruit (piñón) and hunting endure over time [77,78].

Environmental history shows that since 1550 CE, with the arrival of the Spaniards in the territory occupied by the Mapuche people, a colonization process began in which the high-altitude ecosystems were used by the Mapuches as shelter during wars and skirmishes against the Europeans (16th-early–19th centuries) and, in turn, as grazing areas for introduced Euro-Mediterranean livestock [79–81]. Since the end of the 19th century, the timber exploitation of alpine forests has enabled the construction of settlements in the valleys and the construction of a large-scale economy based on wheat cultivation [82–84].

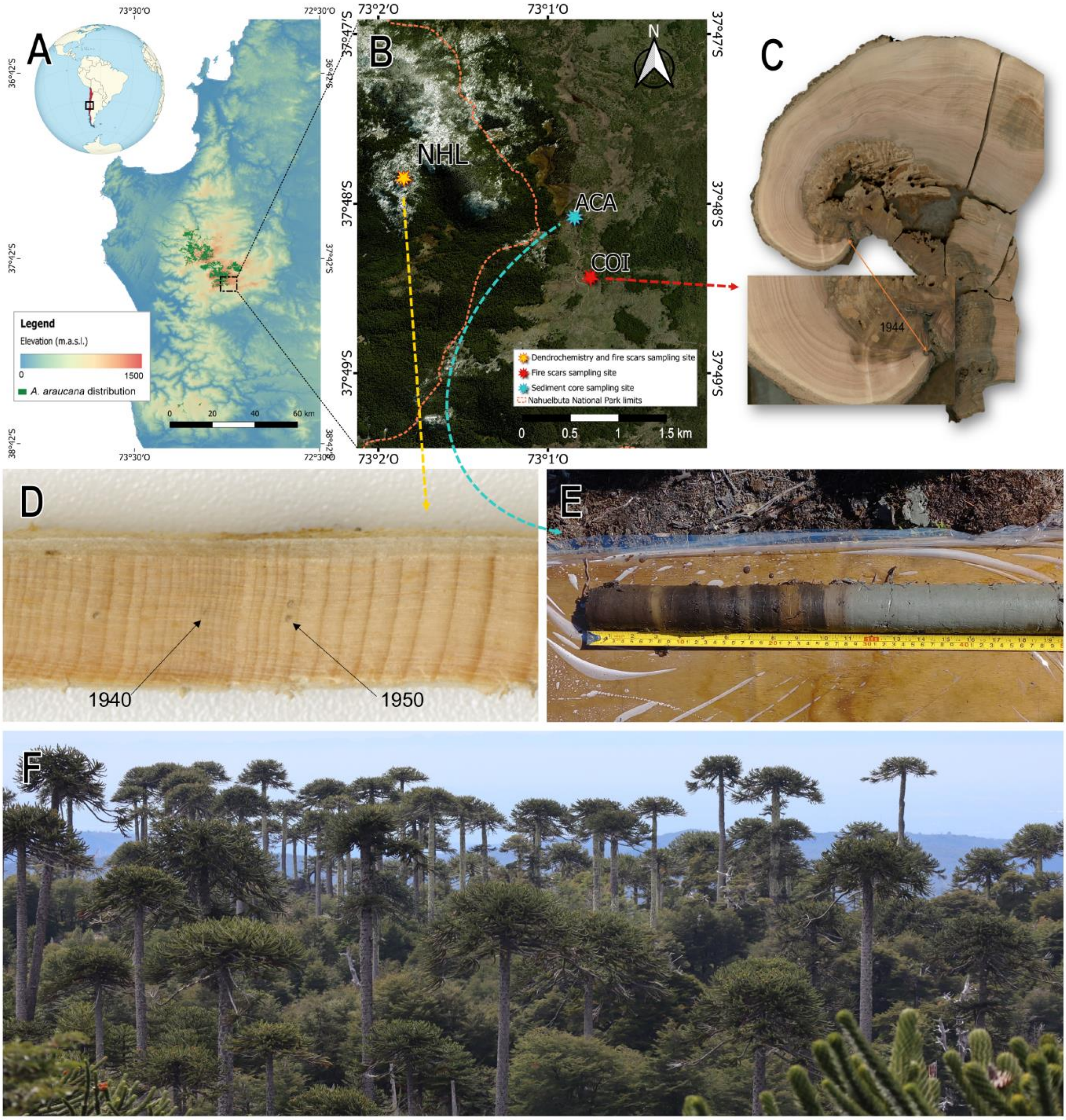

Figure 1. (**A**) Study area: the entire extension of the Nahuelbuta Coastal Range is shown, along with the distribution of *Araucaria araucana* in this area. (**B**) Sampling locations for the different methodological approximations: dendrochemistry (NHL), fire scars (NHL, COI) and charcoal record (ACA). (**C**) Scar sample. (**D**) Segment of processed tree ring core. (**E**) Sediment core. (**F**) *A. araucana* landscape in the Nahuelbuta Coastal Range.

2.2. Dendrochronology Field Sampling

Dendrochemistry: A total of 28 *Araucaria araucana* trees were sampled in April 2021 from the Nahuel site in the Nahuelbuta Coastal Range (NHL, Figure 1B). From each tree, two cores were extracted using 5 mm increment borers (https://haglofsweden.com/project/increment-borers/ (accessed on 30 March 2023)). The chosen trees were healthy, had no fire scars and were growing in shallow soil or rocky substrate. Additionally, data on the diameter at breast height (DBH) and a GPS point at each tree were taken.

Fire scars: Forest patches in the two sampling sites (Figure 1A–C) were intensively searched for fire-scarred *A. araucana* and *Nothofagus* sp. trees and, whenever possible, samples were collected in clusters of several trees to improve the chances of obtaining the most complete fire record possible and increase the likelihood of precisely dating the fires [85]. On standing or fallen dead trees, cross-sections of single and multiple fire scars were extracted with a chainsaw (Figure 1C). For live trees, 2 to 4 cores were extracted close to the fire scar with an increment borer to determine fire dates [86]. In addition, the species, DBH, number of visible fire scars, scar height and azimuth of the scar face and GPS point of each tree were recorded. In total, for the two sampling sites, COI and NHL (Figure 1), 10 and 12 fire-scarred trees were collected, respectively.

2.3. Tree Ring Sample Preparation, Dating and Measurement

Following standard dendrochronological procedures, tree cores and cross-section samples were left to dry, then mounted and sanded with sandpaper of different granulometry (100 to 1000 units) [87,88]. Tree ring samples were then visually cross dated using a stereomicroscope, following the Schulman convention for the Southern Hemisphere [89]. Ring width was measured using a Velmex measuring system (±0.001 mm) (https://www.velmex.com/Products/Pre-configured_Systems/Tree_Ring_Measuring_System.html (accessed on 30 March 2023), and ring width series cross dated using COFECHA software to ensure correct dating and measurement [90]. Ring width series were standardized by applying a negative exponential curve to remove age-related growth effects while building a tree ring chronology for the site using ARSTAN software [91]. This tree ring chronology served as a reference series to precisely date the fire scars of dead tree samples. These scars were identified by the characteristic formation of a cambial death lesion and the healing patterns of radial tree ring growth (Figure 1C) [42].

2.4. Climate-Growth Analysis

In order to identify relationships between climate variables (precipitation and temperature) and tree growth, the resulting chronology NHL was compared with precipitation values obtained from the TerraClimate data set [92] for the period 1958–2020 and mean temperature retrieved from KNMI Climate Explorer (https://climexp.knmi.nl/start.cgi (accessed on 6 November 2022), available for the period 1950–2020. Correlation coefficients were calculated for moving periods, from 1 to 12 months, starting from the second previous growing season to the current dendrochronological year in the Southern Hemisphere.

2.5. Chemical Measurements

The growth series from living trees that made up the NHL chronology (Figure 1D,F) with the best crossdating statistics were selected for chemical measurements in order to develop a dendrochemical chronology for the period 1351–2020. Due to the small size of the Araucaria tree rings (Figure 1D) and minimum mass requirements for chemical analysis (50 mg of sample), this was performed by pooling two consecutive annual rings for the entire period and by creating a composite chemical chronology of selected series. Although the individual signal of each tree is lost with this approach, it allows obtaining a common signal of the group of trees, as well as reducing costs and covering longer periods of time [62]. Thus, we selected 32 tree ring series that jointly covered the entire period under study to obtain a 2-year resolution chemical chronology.

Under a binocular microscope, each core was cut every two years using a sterile non-metallic, ceramic knife over an acrylic table. In order to avoid possible cross-contamination, all segments were then polished with a diamond tool to remove all the surface that was in contact with extraction and sanding tools. Isopropyl alcohol was used to clean all materials. All 2-year samples from the 32 series were then placed in labeled tubes to form composite samples for each biennium (Figure 2A), obtaining a total of 335 samples covering the

1351–2020 period. Processed samples were subsequently sent to the Arizona Laboratory for Emerging Contaminants (ALEC) at the University of Arizona for chemical measurements (https://www.alec.arizona.edu/ (accessed on 15 March 2022)) (Figure 2).

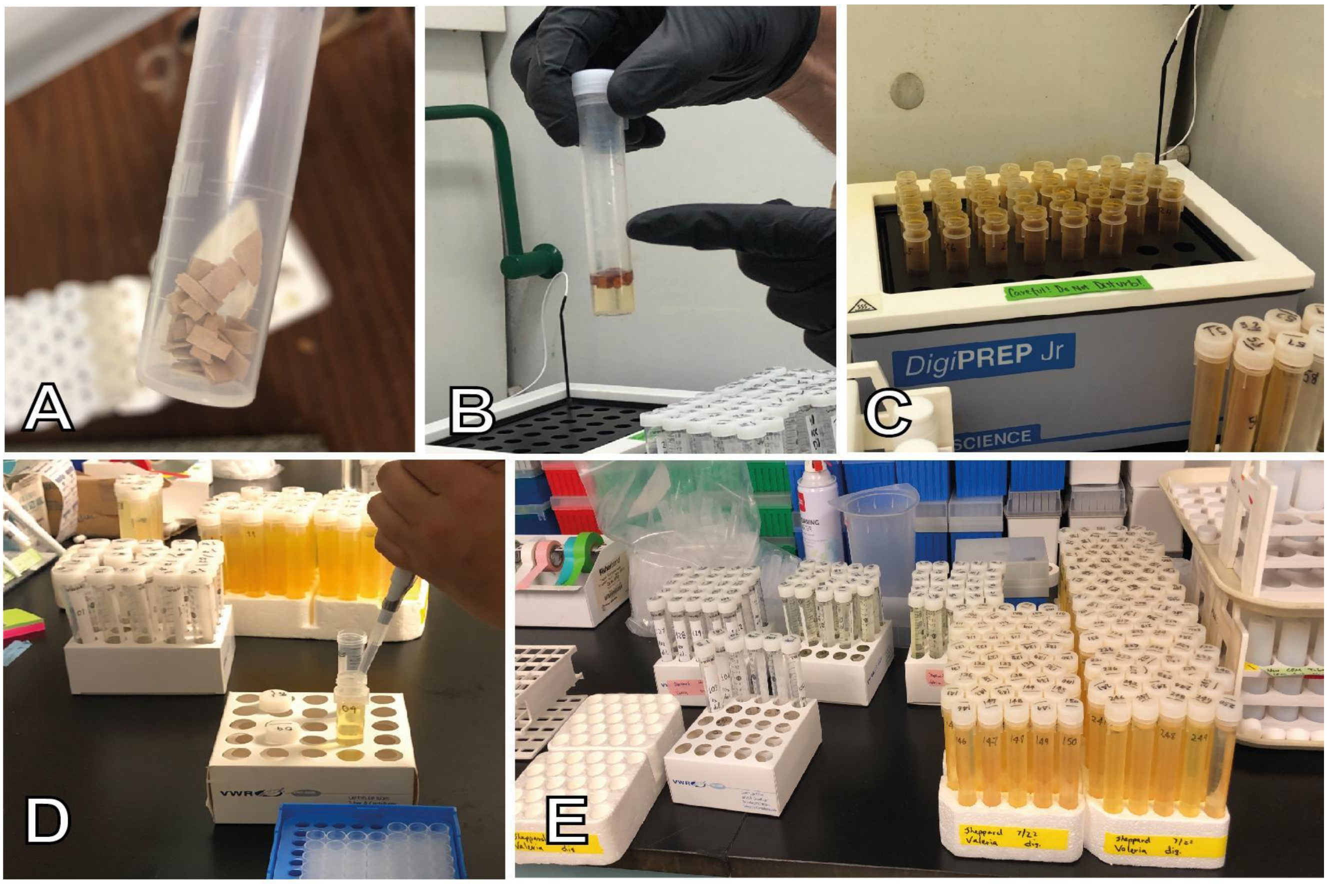

Figure 2. Dendrochemical processing: digestion and dilution of wood samples in Arizona Laboratory for Emerging Contaminants (ALEC). (**A**) Composite sample. (**B**) Acid predigestion of composite sample. (**C**) Dilution of 1.5 mL aliquot of digestate with Mili-Q water. (**D**) Samples in a digestion block to complete digestion. (**E**) Samples ready to be analyzed by ICP-MS.

The samples were prepared via acid digestion. Wood samples were weighed in precleaned, pre-weighed, trace metal-free polypropylene centrifuge tubes. Samples were then predigested with 3 mL of concentrated Optima grade nitric acid (HNO_3) at room temperature for 24 h and then heated at 90 °C in a digestion block for 2 h to complete the digestion (Figure 2B). Sample tubes were then reweighed to calculate exact dilution factors. A 1.5 mL aliquot of digestate was gravimetrically diluted by a factor of approximately 10 with ultrapure 18.2-MΩ/cm water from a Milli-Q system (Merck, Darmstadt, Germany) (Figure 2C,D). The liquid samples (Figure 2E) were analyzed by an inductively coupled plasma mass spectrometry (ICP-MS; 7700) system (Agilent, Santa Clara, CA, USA). Major cations (potassium (K), calcium (Ca), sodium (Na), magnesium (Mg)) and trace metals (beryllium (Be), aluminum (Al), manganese (Mn), nickel (Ni), copper (Cu), zinc (Zn), molybdenum (Mo), silver (Ag), cadmium (Cd), tin (Sn), antimony (Sb), barium (Ba) and lead (Pb)) were measured, along with standards (Pine Needles (SRM 1575a) and one blank sample. The calibration of ICP-MS was performed using Multi-element Standard Solution of Spex CertiPrep (Metuchen, NJ, USA) as well as an independent quality control check from High-Purity Standards (Charleston, SC, USA). Results were expressed as $\mu g\ g^{-1}$ of dry weight of wood.

2.6. Dendrochemical Data Analysis

The biennial time series of chemical element concentrations recorded in *A. araucana* tree rings (Figure S1A,B) were individually detrended and standardized using a spline curve with a 50% frequency cutoff at 70 years in order to remove the magnitude effect of the chemical concentrations and to be able to compare the time series concentrations between them. Superposed Epoch Analysis (SEA) was used to determine the relationship between fire events and chemical element concentrations in the years before, during and after the event. The mean values of these data were calculated for windows of 4 biennia, i.e., 8 years, including the biennium of the fire event. The mean values of the standardized chemical series during the years of the fire event were compared with variations in the entire record by performing 1000 Monte Carlo simulations that randomly select years, calculate expected averages and provide 95% confidence intervals [93,94]. In each case, the number of randomly selected years is equal to the number of actual fire years. In this way, we compare the chemical signals to a subselection of fire dates recorded on at least two site trees from the sampling sites. All statistical analyses were conducted using R [95], RStudio [96] and the dplR package [97,98].

Finally, in order to propose new fire dates prior to the records obtained from fire scars, we selected the chemical elements that showed a clearer response to the fire from the Superposed Epoch Analysis. The proposed fire dates were selected as those in which at least 3 elements of the subset of previously selected elements had a positive chemical pulse above the 90th percentile, either during the same biennium or after two biennia. In addition, we developed a Principal Component Analysis (PCA) for eight selected elements (Figure S2) according to the SEA results to compare with carbon records from sediment cores. In addition, we used a temperature [99] and PDSI [100] reconstruction to compare the fire record estimate from dendrochemistry and charcoal analyses.

2.7. Charcoal Record

Two sediment cores were collected during January 2021 from Aguas Calientes (ACA) peatbog using a Russian corer (Figure 1E). Sediments were described and dated using 14C. Age–depth model performed by Bayesian analyses in "rPlum" package [101,102] in R platform [95]. Six radiocarbon dates (bulk, plant tissue, charcoal) were analyzed in the University of Arizona LTRR and AMS Laboratory and calibrated with the SHcal20 curve [103] (Figure S3). One date was not included in the age–depth model due to its chronostratigraphic error (Table S1).

To infer fire regime changes at Nahuelbuta National Park we used macroscopic charcoal particles (>125 µm for fire peak detection in a radius of <3 km from coring site; [104]). For macroscopic charcoal counts, 2 cm^3 of sediment was taken at 1 cm intervals for the length of the core, sieved through 125 µm screen and tallied under a stereomicroscope at 10–40× following the methodology outlined by [51]. Statistical analyses were performed to detect fire peaks using CHARanalysis software [105]. For that purpose, we interpolated samples at 20 year intervals and calculated the background component using a moving average window with a width of 1000 year and a threshold ratio value of 1.1 to identify fire episodes above background.

3. Results and Discussion

3.1. Fire History of Araucaria-Nothofagus Forests

The fire history of the two sampling sites (NHL and COI) was reconstructed from 19 cross-dated samples, recording 18 fire years in nearly 500 years. Most fire-scarred samples (58%) recorded a single fire scar, while 26% recorded two–three fire scars. The fire chronology developed in COI spanned 468 years, and NHL spanned 478 years. The oldest dated fire was in 1738 and the most recent one occurred in 1981 (Figure 3). The frequency of fire in the last three centuries has increased; in the 18th, 19th and 20th centuries, four, five and nine fire dates, respectively, have been recorded from at least one tree (Figure 3). In this record, seven fires were documented by at least two trees; the majority of those events (five fires) occurred after 1900 (Figure 3).

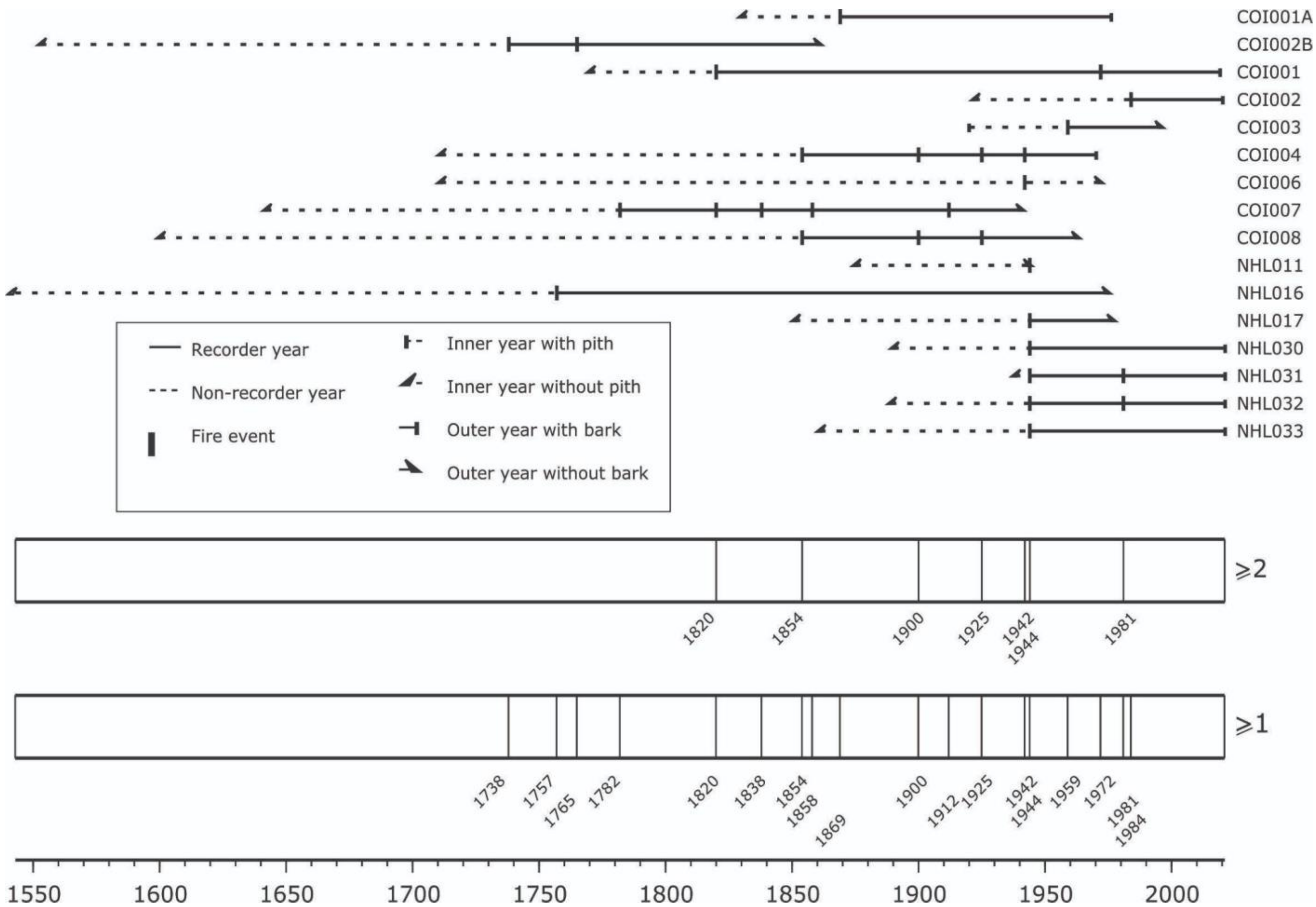

Figure 3. Fire-scar chronologies from two sites in Nahuelbuta Coastal Range: COI and NHL. Horizontal lines represent individual trees and fire-scar events are shown by short vertical lines. Fire events and dates (≥1 or ≥2 fire-scarred trees) are indicated below next to the *x*-axis.

The patterns found in these two sites are like other studies carried out in Araucaria forests in Chile and Argentina. In the Andean Araucaria distribution, during the period of the Euro-Chilean settlements, fire frequency increased significantly [18,21,22,106]. Perhaps counterintuitively, the establishment of protected areas such as the Nahuelbuta National Park, established in 1939, and the Malleco National Reserve, created in 1907, have not reduced fire frequency in these ecosystems. Only after the creation of the National Forest Service (CONAF) in the 1970s, and the implementation of a fire suppression policy, did fire frequency in these protected areas diminish [107]. A similar result was also observed in Tolhuaca and Villarrica National Parks [18,21]. On the other side of the Andes Range, in Argentina, more effective fire suppression policies were implemented in Araucaria forests after the creation of protected areas [22]. Despite the differing fire management strategies on either side of the border, some fire years during the twentieth century, such as the 1944 and 1959 fires at the Nahuelbuta sites and periods of high fire frequency in 1890–1900 and 1760–1770, have also been found in other Araucaria forests in the Chilean and Argentine Andean distribution, suggesting a climatic link between some of these fires [18,22]. On the other hand, the fires identified in the mid-nineteenth century could be related to the recurrent clashes in this area between the military and Mapuche people, who refused to incorporate their territory into the Chilean State [108]. The Nahuelbuta Mountain Range was very important from a military strategic point of view as it constituted a natural boundary from the settlements, and its thick forests and surrounding areas provided refuge for Mapuche people. In the clashes between indigenous people and the Chilean military, the use of fire was a constant (fires in agricultural fields, pastures and settlements in general) and was at its peak between 1852 and 1883, which may partly explain the occurrence of fires in this area, considering that the dates of the fire scars do not coincide with generalized climatic events [108–110].

3.2. Ring Width Chronology of Araucaria araucana

The Ring Width Index chronology of the NHL site used to correctly date fire scars included a total of 28 trees of *A. araucana*, spanning 921 years for the period 1100–2020 with an interseries correlation of 0.501 (Figure 4A). This correlation value is similar to those seen in other chronologies of this species [22,111,112]. The biggest increases in tree growth were observed in 1414 and between 1956 and 1958, while its lowest growths were observed in 1627 and 1896. A very noticeable feature in this chronology is the positive growth trend between the years 1946 and 1956, which is the most important in the whole chronology (Figures S1 and 1D). This release was recorded in most of the trees and appears to be peculiar to this particular site when compared with other chronologies of the species in Nahuelbuta National Park (Figure S4) and seems to be related to fire occurrence (Figure 4A) and logging activities documented in this area for this period. At the NHL site, we also found an increase in variability and releases at the beginning of the chronology, around 1400–1550, which were not associated with decreases or important variations in sample size (Figure 4A).

The growth patterns of *A. araucana* in the NHL site show a positive correlation with the precipitation values of the summer prior to ring formation and also with the current growing season. Growth shows a negative correlation with temperature in the spring and summer of the current growing season (Figure 4B). The highest values of correlation between ring width and a climatic variable at NHL were with accumulated precipitation between past November and current January and with the average temperature of August to October in the current year (r = 0.33 (1958–2020) and r = −0.31 (1950–2020)) (Figure 4B). This climate–growth relationship follows the regional and also local tree-growth patterns shown by [111,112] for the Araucaria population across the distribution of *Araucaria araucana* in Chile and Argentina, including the Piedra del Aguila and Pichinahuel sites, also located in the Nahuelbuta National Park. The Nahuelbuta National Park Araucaria ring width chronologies are some of the longest in the Araucaria network in Chile and Argentina. A SEA using the ring width chronology and selected fire dates revealed negative growth anomalies one year before the fire events (Figure 4C), suggesting that dry conditions, resulting in the formation of small rings, are likely to promote fire events.

3.3. Dendrochemistry and Fire Scar Dates

The concentrations of various chemical elements were measured and were used to determine if there was a relationship with the other fire study proxies, such as fire scars and charcoal in sediments. Figures 5 and 6 show fire scar dates and the detrended and standardized chemical time series since 1700 for all chemical elements analyzed except Be, which was not detected in any sample. The responses of the chemical elements to fire events varied between elements and fire dates recorded in two or more trees. By comparing the dendrochemical data set with these fire dates (1820, 1854, 1900, 1925, 1942, 1944, 1981), we can see that:

1. Some elements increased in concentration during the year of the fire or 2 to 8 years after the fire dates. Positive anomalies (defined as concentration values of >=5 elements above the 90th percentile) were observed in 11 elements (Ba, Ca, Mg, Mn, Cu, Na, Ni, Al, Mo, Pb, Zn and Sb) for the biennial period 1947–1948 (Figures 5 and 6), after the fire events recorded at the NHL site in 1942 and 1944, which could be affecting the chemical responses of elements in tree rings at the site. An increase in tree growth was also observed in the NHL site for this period (Figure 4A). For the fire recorded in 1900 at the COI site, increasing

peaks of Cu, Na, Ni, Al and Pb were found in the same biennial period of the fire. On the other hand, the fire that occurred in 1820, also at the COI site, showed positive anomalies two biennial periods after the fire events in Ba, Ca, Mg, Mn, Ni and Zn. Nevertheless, we could not find significant positive anomalies for more than five elements at the same time for the fires occurring in 1854, 1925 and 1981 (Figures 5 and 6).

2. A few elements increased their concentrations in the years prior to fire events. This is true for the year 1900, where Cu, Na, Ni, Al and Pb increased their concentration 1 biennium prior to the fire date. Other fire dates that show this behavior in trace elements are 1912 (same elements as 1900), 1869 (Ba, Ca, Mg, Cu and K) and 1738 (Ba, Ca, Mg, Mn, Cu, Ni and Mo), corresponding to fire dates detected only in one tree.

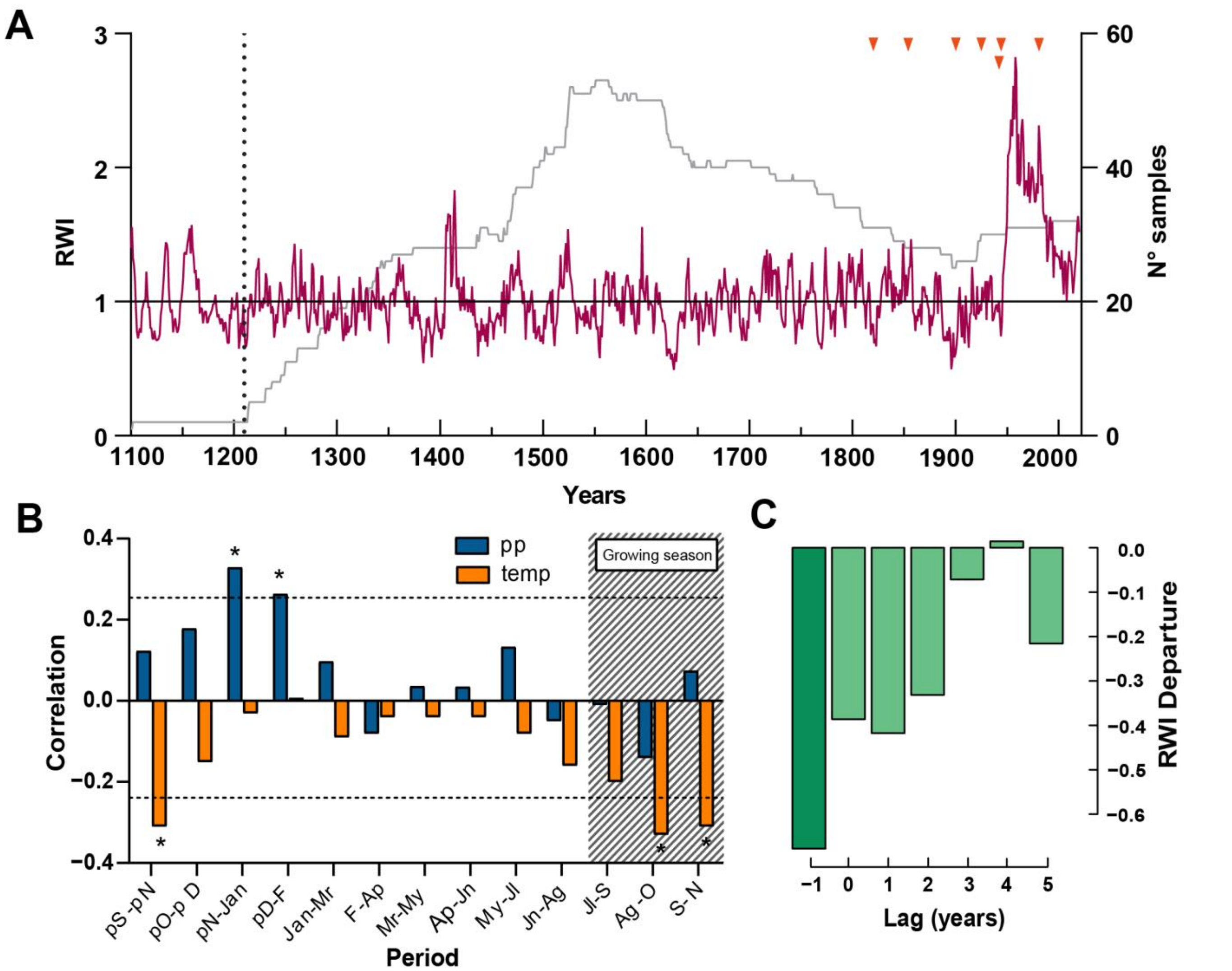

Figure 4. (**A**) Ring width index chronology (RWI) of NHL site for the period 1100–2020 (fuchsia line). Vertical dotted line indicates where EPS > 0.85. Right vertical axis shows the number of samples (gray line). Orange triangles indicate years where ≥2 trees presented fire scars. (**B**) Correlations between accumulated precipitation (blue, 1958–2020) and average temperature (orange, 1950–2020) in periods of three months with the RWI chronology. Horizontal dashed lines indicate critical r values and asterisks indicate significant correlations. (**C**) Superposed Epoch Analysis between RWI chronology in the period 1350–2020 and fire dates registered in ≥2 trees. The *x* axis represents a 7-year period, from 1 year before the fire to 5 years after the fire. The dark-green colored box indicates a 95% confidence level for the analysis.

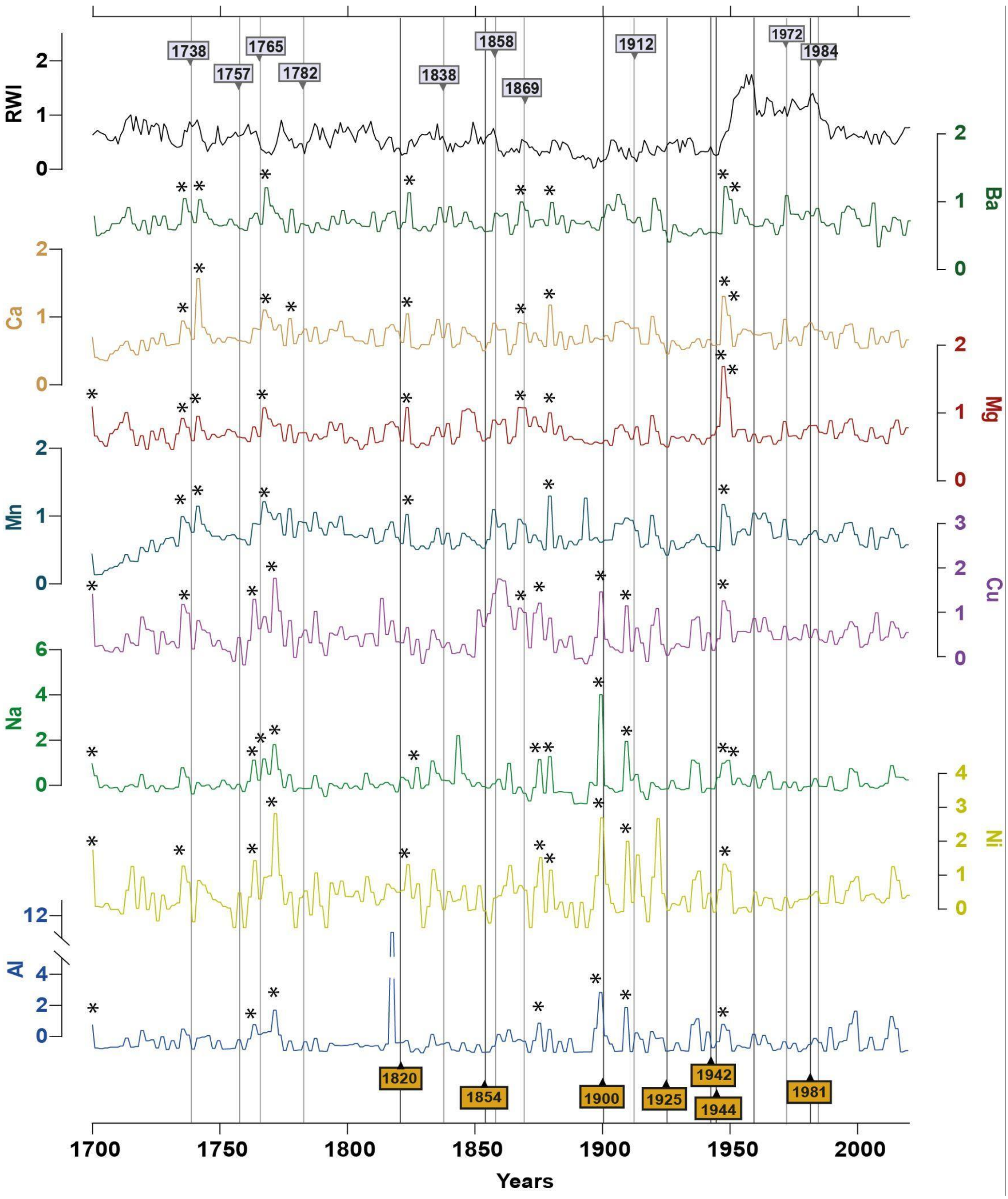

Figure 5. Standardized series of element concentrations in tree rings over time. Vertical lines extending from the x-axis indicate fire events recorded by fire scars in 1 tree (gray) and are shown in the upper x-axis, and ≥2 trees (black) are shown in the lower x-axis. Due to the extent of scar-dated events, the chemical record is shown from 1700 to 2020. Asterisks (*) indicate elements above p90 when ≥5 elements are over p90. RWI = Ring Width Index.

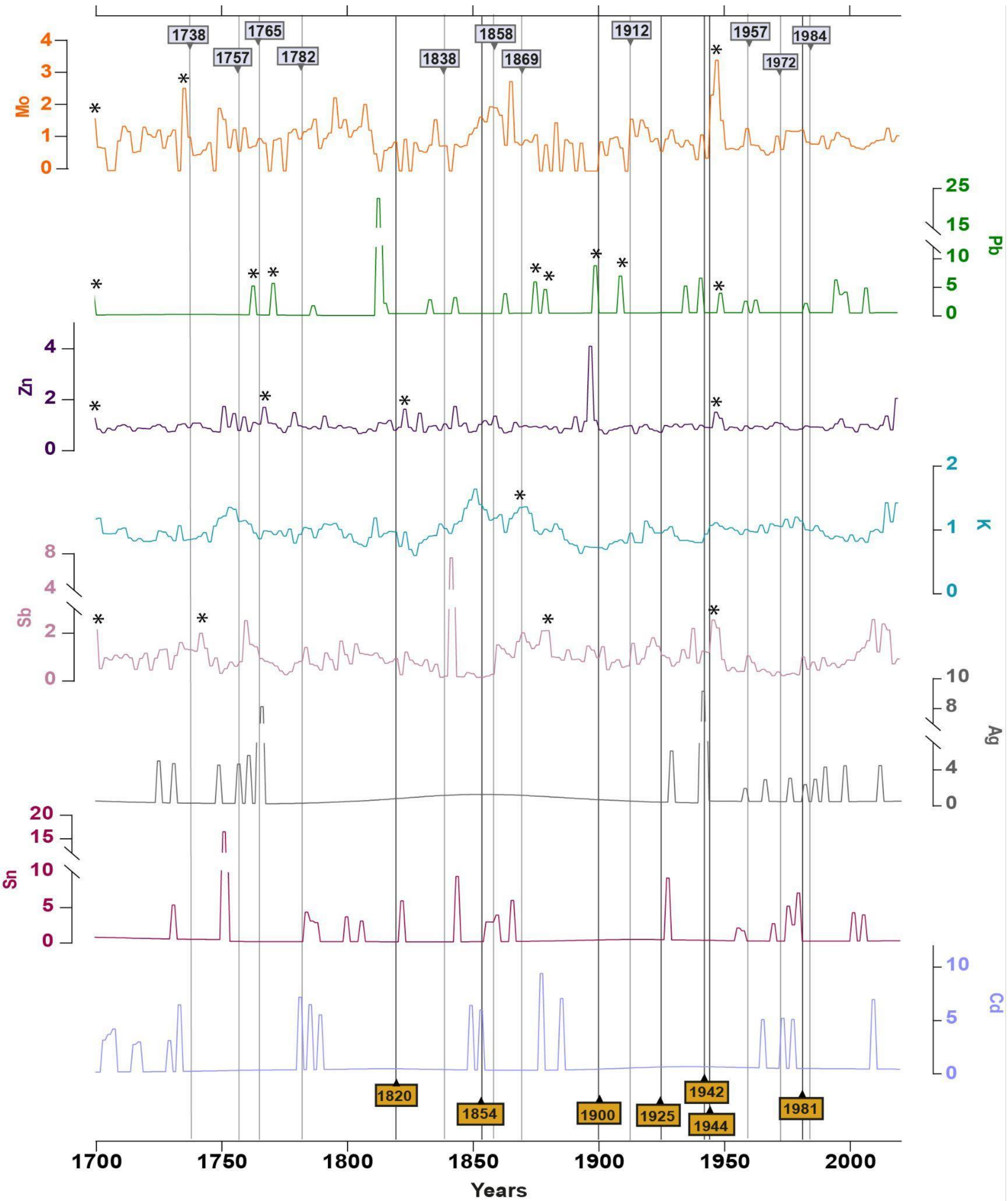

Figure 6. Standardized series of element concentrations in tree rings over time. Vertical lines extending from the *x*-axis indicate fire events recorded by fire scars in 1 tree (gray) and ≥2 trees (black). Due to the extent of scar-dated events, the chemical record is shown from 1700 to 2020. Asterisks (*) indicate elements above p90 when ≥5 elements are over p90. RWI = Ring Width Index.

The SEA carried out to assess the response pattern of significant changes in chemical elements related to the fire events recorded by two or more trees (1820, 1854, 1900, 1925, 1942, 1944, 1981, Figure 3) showed positive significant ($p > 0.05$) anomalies for Mg, Ca, Sn, Mo and Ba after the fire dates for up to 3 biennia (Figure 7). Sn ($p > 0.05$), Ag ($p > 0.05$), Mn ($p > 0.05$), Ba ($p > 0.05$), Na ($p > 0.1$) and Pb ($p > 0.1$) showed positive significant anomalies in the same biennial period of the fire event (Figure 7). On the other hand, Al and Zn showed positive anomalies a two-year period before the fire ($p > 0.05$), as well as Ag ($p > 0.1$) (Figure 7). The remaining elements (i.e., K, Ni, Cu, Cd and Sb) did not show significant anomalies related to fire events (Figure 7).

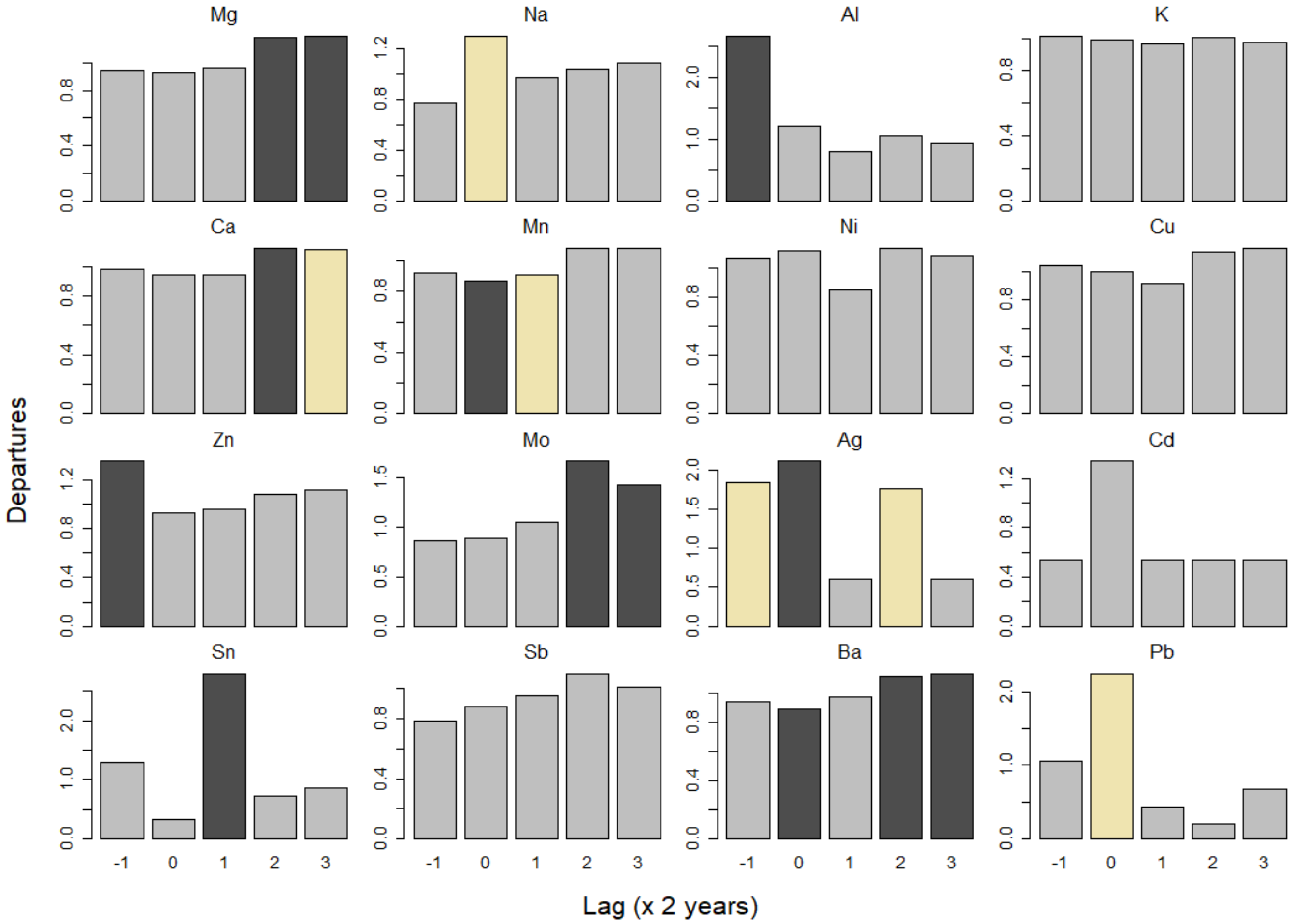

Figure 7. Superposed Epoch Analysis (SEA) comparing standardized "departures" of element concentrations contained in biannual portions of *A. araucana* rings, with fire dates recorded in two or more trees in the period 1700–2020. The *x*-axis represents 5 biennia, equivalent to 10 years, from 1 biennium before the fire event to 3 biennia after. The black bars and yellow bars represent significant confidence levels at 95% and 90%, respectively.

Positive anomalies in chemical series and SEA somehow demonstrate that the main response of most elements in tree rings after fire is an increase in concentrations following fire events. It is widely known that fire remobilizes trace elements in the environment, increasing their concentration in soils and sediments [63,64]. Through the uptake, discrimination and fixation mechanisms [65] can be different; these trace elements can be incorporated into the wood tissues during the same year of growth or be delayed by a few years because of differential incorporation rates [65]. Furthermore, trace elements have been shown to remobilize and translocate between tree rings [53,113]; this could also be an explanation for why some elements show increases in the years prior to the fire events. Another explanation for prior chemical increases could be fire dates potentially were not captured by the fire scar sampling in Nahuelbuta forest, especially because currently it is not permitted to take fire scar samples from living trees, as has been specified previously for other Araucaria forests. This could explain why we did not find fire records around the end of 19th century that were found in other fire histories of Araucaria forests in Chile and Argentina [18,22]. On the other hand, a study conducted in an Araucaria-Nothofagus forest in the Tolhuaca National Park in the Andes Mountains, affected by a high-severity fire in 2002 [114], found that there were elevated concentrations of Mg, Ba, Mn and Ca three biennia before the fire related to the presence of cells occluded with phenolic compounds, typical of the effect of chemical compartmentalization. This chemical response coincides with those found by [66,67] near the healing callus where these occlusions occur as resin soaks before and after fires [115]. However, in the current study, we tried to avoid erroneous indicators of possible fire events from the Araucaria dendrochemical records by: (a) selecting only trees without fire scars, and (b) selecting fire dates where at least three or more chemical elements showed a clear increase in the chemical concentration, which is best explained in Section 3.4 of this study.

Some elements in tree rings have been proven to record chemical changes associated with natural or anthropogenic activities in the same site and also close to the site where the trees came from [56,62,116]. In the case of our dendrochemical results in the Nahuelbuta Range, we found an interesting match between the increase in concentrations of several elements in the Araucarias of the NHL site and the presence of fire scars there and in a nearby site (COI) as well as with carbon abundance from nearby lake sediments cores (Figure 1A,B). Dendrochemical records integrate chemical changes in the local area and also a wide geographical range surrounding the sample site and its environment. It is possible this is due to the potential of wildfires to mobilize chemical elements in ecosystems that are then spread by smoke far downwind [64]. However, several fires were not detected by concentrations of elements in tree rings. A possible explanation could be a low fire intensity or severity. It is highly probable that more severe fires are more easily detected in dendrochemical records than small ones because of the magnitude of remobilized trace elements in the environment [63]. Another explanation for the variation in element concentrations in trees that survive fire could be explained by variability in which types of elements are mobilized in each individual fire [66,117]. It is known that fires significantly increase nutrient availability, such as Ca, Mg, K and Na, associated with ash deposition and the combustion of organic matter, which increase their concentrations in soils with increasing fire intensity [118]. However, these increases in concentration of exchangeable cations can be short-lived in soils due to the high volatilization and evapotranspiration rates associated with new recruitment and remnant plants after fires [118]. Furthermore, [119] analyzed the concentrations of several trace elements in soils and ash after forest fires in eucalyptus and pine plantations in Portugal, finding high levels of V, Mn, Ni, Cd and Pb in burned soils, while Co and Cu concentrations did not change after fire. Even though increases were reported following fire events, some of them (i.e., Mn and Cd) showed fast reductions after the first rain events, while V, Co and Ni increased through the first eight months after the fire event, and Cu and Pb showed small changes through time. Soil acidity also plays an important role in controlling element availability for plants and other ecosystem processes. Ref. [120] reported Ca, Mg and Mn were significantly correlated to the acidity status of the soil. They suggest that if the elements correlate significantly to a common factor, then they might also correlate with each other. This also can explain the common signal between some elements after fires in the Araucaria Nahuelbuta forest.

Dendrochemical studies have been widely used to reconstruct trace metal deposition from various pollution sources, and its use in dendrochemical monitoring seems promising [55]. There is evidence that the chemical composition of wood is affected by fire, showing changes in element concentrations surrounding fire scar samples [60,66,67], as is the case for the present study. Hence, it may be possible to use some of these elements found in this study to extend the fire history of the Nahuelbuta Coastal Range based on tree ring dendrochemical records. However, the chemical responses to fire dates were not always the same, so the interpretation of these conclusions and those of dendrochemical studies in general should be cautiously applied [121] due to the recurring dendrochemical uncertainties described over the years by several authors (e.g., [53,54,113,121]). These are, but are not limited to: (1) the variety of uptake paths for chemical elements, uses and movement within the xylem and the trunk [53,121], (2) the variability between trees, sites and species [67,122], (3) the climate and environmental conditions potentially related to the uptake of some chemical elements [123,124] and (4) the age of the trees, structure and composition of the forest and canopy [125] and the pH of soil, which play a significant role as well [54].

3.4. Extending the Fire History of Nahuelbuta Range Using Dendrochemistry and Charcoal Records from Sediment Cores

Using a set of chemical elements with pulses of concentration over the 90th percentile, which also match with three other elements in the same condition, we propose a new set of fire periods (Figure 8). The new fire dates proposed include, in most of the cases, chemical elements considered relatively less mobile in the trunk, and avoiding groups only represented by chemical elements described as easily mobile (K, Mn, Cu, Zn) [72]. From this analysis, at least three periods where various elements presented pulses of chemical elements that exceeded their p90 were identified. The first, between 1363 and 1404, presents pulses in all its elements at least once, except Na. The second period extends between 1455 and 1552, and a third period between 1601 and 1700, with concentration pulses of all chemical elements above their 90th percentile (Figure 8). These results suggest the occurrence of periods of greater fire activity in the past than in the calibration period of scars and fires (1700–2020; Figure 8).

These results were also compared with regional climate reconstructions and charcoal records from sediment cores obtained in nearby lakes to the dendrochemical site and the fire scars. This analysis suggests that there are important coincidences between periods of greater presence of carbon in sediments (Figure 9A), periods of high temperatures and dry conditions (Figure 9B), where there are also pulses of chemical elements in Araucaria trees (Figure 9C), demonstrating the consistency of these records and corroborating the influence of the climate on the occurrence of fires. This information constitutes the first time that dendrochemical records calibrated with fire scars have provided information to reconstruct fire regimes, which in turn coincide with charcoal records in lake sediments (Figure 9).

The results from Aguas Calientes charcoal records show that the greatest accumulation occurs between the years 1200 and 1650 AD, distributed with three maximums or peaks recorded in the years 1200, 1380 and 1600 AD (Figure 9A). These charcoal particle maximums coincide with warm and dry periods, from regional climate reconstructions developed with tree rings (Figure 9B), and also with increases in the concentrations of chemical elements in Araucaria trees at the NHL site, which shows its greatest activity (higher chemical pulses) between 1400–1650, confirming greater climatic variability in these periods.

These small differences in the timing of fire activity may be associated with the sedimentation rates and chronological control of the Aguas Calientes records; one radiocarbon date of 525 $\pm$ 20 years 14C AP located in the AC2101AT1 core, 33 cm deep, supports the age–depth model for this section of the sediments. This dating is associated with the maximum amount of charcoal registered in the year 1400 AD, which allows us to establish that this moment was the largest fire registered in Nahuelbuta in the last thousand years. It should also be noted that, given the nature of the charcoal data, these fire events are expressed in the sediments as a Gaussian curve, typically caused by the resuspension and reaccumulation of sediments after one fire event. In this sense, wetlands or swamps are excellent sites for the detection of fire events given the little remobilization of sediments that occurs there [126]. More radiocarbon dating in the sediments, and a large number of trees, sites and chemical elements, could improve the statistical relationship between these two proxies (dendrochemistry and charcoal record). Moreover, other paleoenvironmental indicators, such as palynology and geochemical analysis in sediment cores, can also provide most indicators to analyze together.

The beginning of the period with the highest fire activity in Nahuelbuta (around 1200–1450 AD; Figure 9) is also consistent with a period of higher temperatures [99] and lower-than-average precipitation around 1200 CE at 41° S [127]. Thereafter, multi-decadal dry spells were recurrent during the first half of the last millennium (11th, 13th, 16th centuries) in south-central Chile [5], which also supports the case for extended fire activity around 1100–1450 CE. For the Araucaria distribution, warm atmospheric temperatures superimposed with dry years and/or summers are also a keystone in promoting widespread fires mainly driven by the desiccation of dead biomass [18–20].

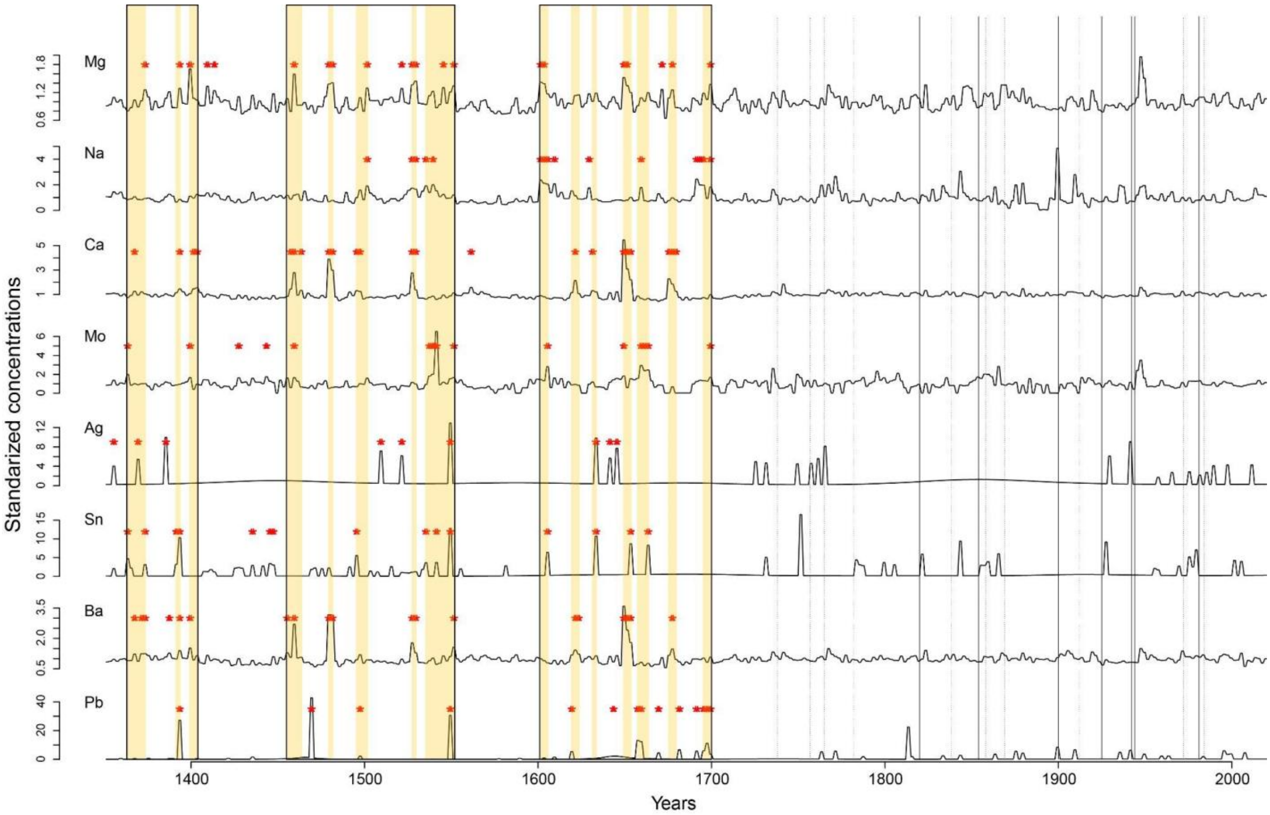

Figure 8. Standardized series of element concentrations in tree rings over time. Vertical lines extending from the *x*-axis indicate fire events recorded by fire scars in ≥2 trees (solid black lines), 1 tree (dotted black lines). Red asterisks (*) show all years in which standardized element concentrations exceed their 90 percentile. Possible fire events or periods are shown in yellow rectangles when ≥3 element concentrations are above p90 at the same biennium or after one or two biennia. Black border rectangles show three periods separated for 51 and 49 years, respectively.

Several authors have shown a correlation between fire years in Patagonia and south-central Chile with droughts derived by large-scale atmospheric anomalies associated with SAM and/or ENSO forcings [17–20,128], including at multi-centennial scale [126,129–132]. Our paleoenvironmental reconstructions show that the major biomass burning period around 1200–1450 AD coincided very nearly with El Niño-like and positive SAM-like conditions [133,134]. Today, the co-occurrence of the positive SAM phase and El Niño events during summers has led to extremely warm and dry conditions across Patagonia and promoted widespread fires on both sides of the Andes [17,22,28,128]. This study, however, is the first time this pattern has been proved in the mid-latitudes coastal areas. On the other hand, the cultural history of the study area is characterized, starting around 1300 BP, by the presence of groups that have a diversified adaptive transition strategy between hunter-gatherers and the first pottery manifestations (Pitren), with high mobility, which allowed them in their seasonal itineraries to access resources from the sea, lakes and mountain lagoons, as well as collect the fruit of Araucaria trees [135]. Further investigation into the anthropic signal associated with the incendiary activities for that period may be possible with information from future archaeological studies in the highlands of the Nahuelbuta Range.

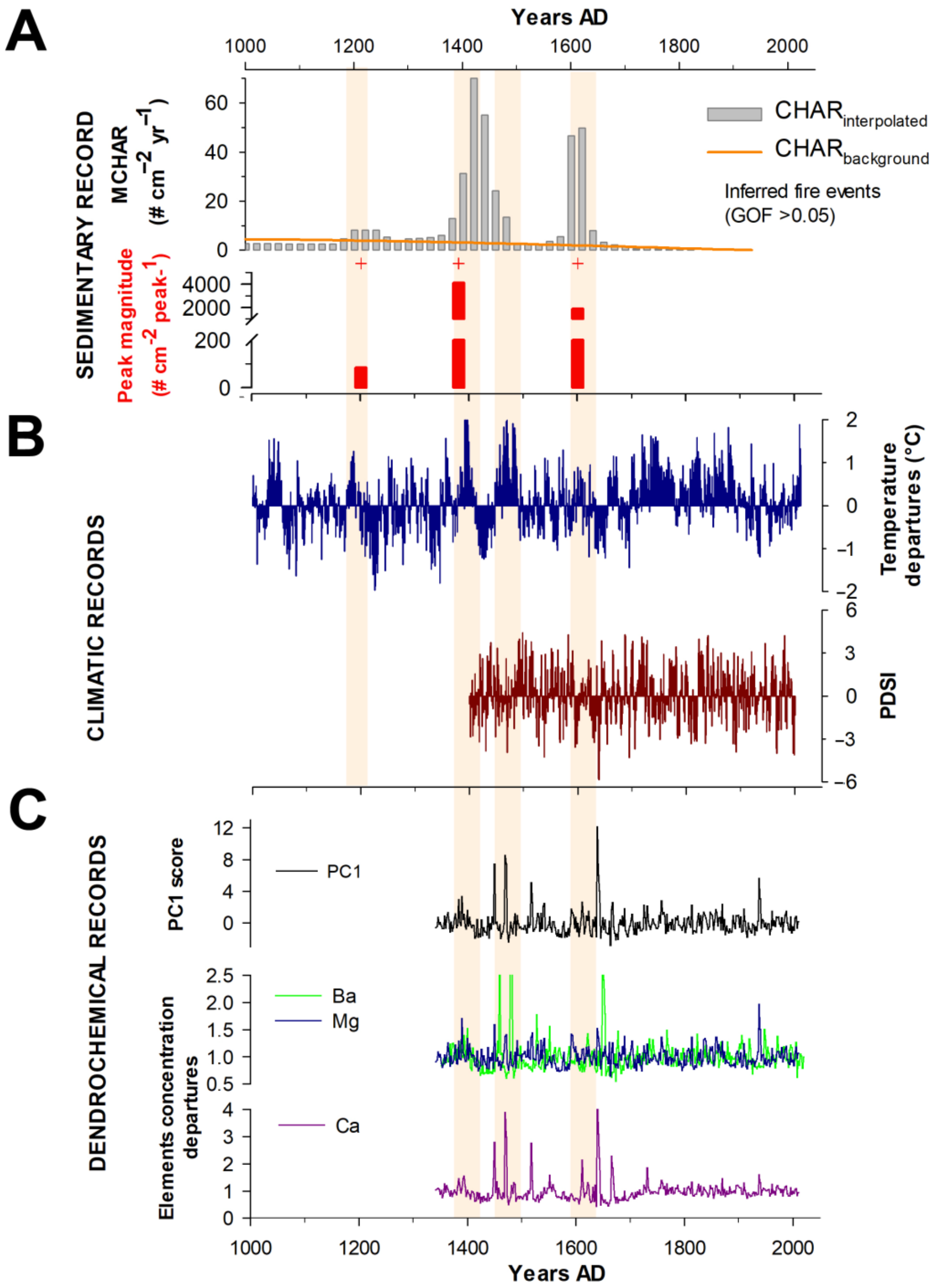

Figure 9. (**A**) Results of CHAR analysis of the Aguas Calientes sediment record for the last 1000 years. In the top graph, the MCHAR (charcoal accumulation rates) values are shown in gray, the interpolated Background curve in orange. The red bars in the lower graph shows the magnitude of the statistically significant fire events detected by the CHAR analysis marked with the red crosses. (**B**) Climatic records from the temperature reconstruction (°C) developed by [99] (blue) and PDSI reconstruction developed by [100] (dark red). (**C**) Dendrochemical records. The upper graph shows the result of the PC1 for eight selected elements according to the results of the SEA (Mg, Na, Ca, Mo, Ag, Sn, Ba and Pb). Ba, Mg and Ca were the elements that contributed the most to PC1 (see Figure S2).

The documentary use of archives, complemented by the analysis of archaeological sites, allows us to understand how the cultural practices associated with the use of fire may have left their mark on the study area in the last 1000 years. Fire management through time in these territories is directly related to use intensification by human communities, both in prehistoric and historical times, and the changes in the economic patterns involved (gathering/horticulture-slash-and-graze agriculture/Euro-Mediterranean livestock farming/strategies of warfare/the installation of new European economies), resulting, thereby, in a mosaic-type landscape in the territory [136,137], which is congruent with cleared areas observed since the 16th century by Spaniards [138].

On the other hand, the maximum of charcoal observed around the year 1600 AD is very interesting (Figure 9A), where the regional climatic trend is associated with higher amounts of precipitation and lower temperatures [99,139], and just a few dendrochemical records match with the driest events in the PDSI reconstruction in the years 1638–1639, but most of their pulses occur in the wet and cold previous period (Figure 9B,C). Because of that, we suppose it could be related to increased human activity in the study area. Historical records from central and southern Chile document that, at the time of the arrival of the Spanish, the Nahuelbuta Mountain Range would have been used as an area of refuge for indigenous communities that were located in lowland areas, such as Lanalhue or the Purén-Lumaco Valley. Since the 17th century, the Mapuche have become an itinerant and mainly livestock-focused society, adopting Euro-Mediterranean animals such as cows, horses and sheep [108,140]. At the same time, the war between the Spanish and the Mapuche accelerated the abandonment of the valleys until the end of the 19th century [141–144].

Most of the observed patterns in this multiproxy study of the Nahuelbuta Range confirm the mixed contribution of both climate and humans to the fire regime through time. Future studies should add more paleoenvironmental records to better understand the fire regime changes in this relictual Araucaria forest.

4. Conclusions

Our data indicate that dendrochemical records can contribute as a proxy of fire activity, complementing fire history studies developed by other techniques.

The main result of this study is the increase in chemical concentration of some elements such as Ca, Mg, Ba among others after fires, which were calibrated with fire scar tree ring data and compared with charcoal records from sediment cores. At the same time, our results also show that dendrochemical approaches require calibration with other proxies and a careful interpretation of chemical patterns. Additional studies of dendrochemical response to fires are also needed to improve understanding of the potential capacities and limitations of these techniques to provide longer records of fire history studies in these forest ecosystems. More information about the chemical mobilization in the trunk from different elements and the chemical characterization of forest composition is needed to best understand sources and concentration changes in this forest ecosystem.

Through the multiproxy approach used in this study, for the first time we presented a millennial high-resolution fire history. These results show that an apparent higher fire activity occurs in the period 1200–1650, especially after the years around 1200, 1380 and 1600. On the other hand, a progressively increasing frequency of low-severity fire was also observed after 1700, concomitant with other Araucaria fire histories developed for the Andean range in Chile and Argentina. This information creates a new perspective about fire temporal patterns before and after European colonization in the coastal mountains of south-central Chile.

Finally, climate variability and human land use influence forest fire dynamics in Araucaria forests. More information about past human activities, climate and fire relationships before and after the American colonization are needed to understand current landscape changes and their consequences for forest ecosystems.

Supplementary Materials: The following supporting information can be downloaded at: https://www.mdpi.com/article/10.3390/f14061082/s1, Figure S1A,B: Raw element concentrations derived from dendrochemical analyses in the NHL site; Figure S2: Result of Principal Component Analysis (PCA) to be compared with carbon records, temperature and PDSI reconstructions. The elements that contribute the most to PC1 (fuchsia) and those that contribute the least to PC1 (blue) are observed; Figure S3: Age–depth model performed by Bayesian analyses. Six radiocarbon dates (bulk, plant tissue and charcoal) were analyzed and calibrated with the SHcal20 curve; Figure S4: Ring width index chronology of three sites in Nahuelbuta coastal range: NHL (fuchsia), PAG (green), NAH (blue) (The orange vertical line indicates the year prior to release in NHL (1945)); Table S1: Table with dates used in age–depth model. One date was not included in the age–depth model due to its chronostratigraphic error, marked with an asterisk (*).

Author Contributions: Conceptualization, A.A.M., M.E.G. and A.M.A.; methodology, P.R.S., K.K.-B., I.S.-V., M.M.-B., M.M., C.G.R., V.T.-M., B.P., A.M.-C., B.V., A.A.M., M.E.G., A.M.A., M.E.S. and T.R.; formal analysis, I.S.-V., M.M.-B., K.K.-B., C.G.R., B.P., A.M.-C. and B.P.; investigation, A.A.M., M.E.G., A.M.A. and M.E.S.; resources, A.A.M., M.E.G., A.M.A. and M.E.S.; writing—original draft preparation, I.S.-V., A.A.M., M.M.-B., V.T.-M., M.E.G. and K.K.-B.; writing—review and editing, A.A.M., M.E.G., I.S.-V., A.M.A., M.M.-B., K.K.-B., M.E.S., P.R.S. and F.G.; supervision, A.A.M., M.E.G., I.S.-V. and K.K.-B.; project administration, M.E.G.; funding acquisition, M.E.G., A.A.M., A.M.A. and M.E.S. All authors have read and agreed to the published version of the manuscript.

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