

Comparative analysis of the chemical composition and biofuel properties of conifer cone residues from different species

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Abstract: This study aims to investigate the biofuel potential of cone residues from four coniferous species [Taurus cedar (*Cedrus libani* A. Rich.), Turkish red pine (*Pinus brutia* Ten.), Black pine (*Pinus nigra* subsp. *pallasiana* (Lamb.) Holmboe), and Scots pine (*Pinus sylvestris* L.)] from the Isparta region. Cone samples are materials that can be considered waste biomass after seed extraction and have the potential to serve as an alternative source for energy production due to their lignocellulosic structure. The study determined the calorific values, volatile matter, ash, sulphur content, and elemental composition of the samples, and also identified their characteristic functional groups using FT-IR analysis. According to the results obtained, the calorific values of pine cone samples ranged between 3954–5050 kcal/kg and were comparable to wood-based biomass. The volatile matter ratios of 58–64% and sulphur contents of 0.02–0.10% indicated that these materials are environmentally clean fuels. FT-IR analyses confirmed the presence of characteristic O–H, C–H, C=O, and C–O functional groups associated with the lignin, cellulose, and hemicellulose structure in all species. As a result, it has been determined that pine cones, particularly those from the Toros cedar and red pine, are suitable raw materials for biofuel production due to their high energy content and low ash and sulphur content. These findings indicate that pine cone residues can be utilized in renewable energy production, thereby contributing to sustainable forest management and waste biomass recovery.

Keywords: Biomass energy, Calorific value, Cone residues, Coniferous species, Non-wood forest products, Volatile matter

Farklı türlerden elde edilen kozalak kalıntılarının kimyasal bileşimi ve biyoyakıt özelliklerinin karşılaştırmalı analizi

Öz: Bu çalışma, Isparta yöresinden farklı yükseltilerden toplanan dört iğne yapraklı türün [Toros sediri (*Cedrus libani* A. Rich.), kızılçam (*Pinus brutia* Ten.), karaçam (*Pinus nigra* subsp. *pallasiana* (Lamb.) Holmboe) ve sarıçam (*Pinus sylvestris* L.)] kozalak artıklarının biyoyakıt potansiyelini araştırmayı amaçlamaktadır. Kozalak örnekleri, tohum çıkarımı sonrasında atık biyokütle olarak değerlendirilebilecek materyaller olup, lignoselülozik yapıları sayesinde enerji üretiminde alternatif bir kaynak oluşturma potansiyeline sahiptir. Çalışmada numunelerin kalorifik değerleri, uçucu madde, kül, kükürt içerikleri ve elementel bileşimleri belirlenmiş, ayrıca FT-IR analizleriyle karakteristik fonksiyonel grupları tanımlanmıştır. Elde edilen sonuçlara göre kozalak örneklerinin kalorifik değerleri 3954–5050 kcal/kg arasında değişmiş ve odun temelli biyokütlelerle karşılaştırılabilir düzeyde bulunmuştur. Uçucu madde oranlarının %58–64, kükürt içeriklerinin %0,02–0,10 aralığında olması, bu materyallerin çevre açısından temiz yakıt özelliğine sahip olduğunu göstermiştir. FT-IR analizleri, tüm türlerde lignin, selüloz ve hemiselüloz yapısına ait karakteristik O–H, C–H, C=O ve C–O fonksiyonel gruplarının varlığını doğrulamıştır. Sonuç olarak, özellikle Toros sediri ve kızılçam kozalaklarının yüksek enerji içeriği, düşük kül ve kükürt oranlarıyla biyoyakıt üretiminde kullanılabilecek uygun hammaddeler olduğu belirlenmiştir. Bu bulgular, kozalak artıklarının yenilenebilir enerji üretiminde değerlendirilerek sürdürülebilir orman yönetimine ve atık biyokütle geri kazanımına katkı sağlayabileceğini göstermektedir.

Anahtar kelimeler: Biyokütle enerjisi, Kozalak artıkları, İğne yapraklı türler, Kalorifik değer, Odun dışı orman ürünleri, Uçucu madde

1. Introduction

Trees and their components (e.g., wood, bark, cone, needles, root) are one of the oldest raw material for human beings. Although wood-based products have become the primary product of trees, researchers have been interested in the utilization of those residues due to lignocellulosic contents (Vorobev, 2007; Şahin and Yalcın, 2017). However,

conifer cones, which vary markedly between species, such as conic, cylindrical, ovoid, small to large, short to long, etc., are the primary organs that contain the reproductive seeds (Northington and Edward, 1996; Young and Giese, 2002). It has been reported by a number of researchers that cones have similar chemical constituents as wood, which typically consists of cellulose, lignin, and hemicellulose as major constituents, but in different proportions (Baeza and Freer,

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2000; Balaban Ucar and Gonultas, 2014; Fengel and Wegener, 2011; Xu et al., 2012).

Various types of conifer cones have received attention for value-added products. However, recent literature reports on cones have usually focused on chemical constituents which may offer some valuable products for medicinal purposes (Zulaica-Villagomez et al., 2005; Font et al., 2009; Keefover-Ring and Linhart, 2010; Yang et al., 2010). It has already been reported that pine cones are rich in terpenoids and phenolics that are potentially useful as intermediates in the synthesis of scenting agents (Wajs-Bonikowska et al., 2017). Some of the cone extracts were found to be able to cure some disorders (i.e. diarrhea, in cough remedies, help break fever) (Bhardwaj et al., 2021; Malik et al., 2021). Some other types of utilization of conifer cones are also possible, such as creating arts, crafts and decorative elements (Sahin and Yalcin, 2017).

The utilization of biomass rather than wood has been questioned in the environment, mainly concerning its functionality on forestlands. However, after seed extraction, conifer cones have become waste biomass. It has been proposed that the cones are an important indication of fire risk for land management both as fire hazard and refrigeration difficulties (Kwiatkowski and Szejna, 2024; Sahin and Yalcin, 2017). These residues could create conditions not suited to germination by blocking soil and minimizing moisture and light (Northington and Edward, 1996; Vorobev, 2007). Moreover, the management of seed extracted and dried cones is very difficult because their volume increases so that they require considerable storage space (Kwiatkowski and Szejna, 2024).

Biomass energy is one of humanity's earliest sources. The cones from coniferous trees could be potentially utilized as an energy resource (Nicholls et al., 2008; Brack, 2017). It has been suggested that conifer cones are particularly suitable for bioenergy production and that the high lignin content can be

advantageous for energy levels (Picchio et al., 2020). It has been suggested that cones might be a stable and supplementary source of biomass in local utilization (Nicholls et al., 2008; Kwiatkowski and Szejna, 2024).

Some developed countries (e.g., the European Union) have established a new energy policy that would like to increase the share of renewable energy in total energy consumption (Union, 2009; Kwiatkowski and Szejna, 2024). However, solid biomass energy is reported to be the largest share of renewable energy production, which exceeded 40% in the EU. This energy is derived mainly from forests and the timber industry, and it consists of sawmill waste (Kwiatkowski and Szejna, 2024). The aim of this study was to determine the biofuel properties of selected four different conifer cones of Turkish Red pine, Scots pine, Black pine and Taurus cedar cones after seed extraction to evaluate the calorific, burning and gas properties and elemental composition.

2. Material and methods

Cone samples from four widely distributed coniferous species Turkish red pine (*Pinus brutia* Ten.), Black pine (*Pinus nigra* subsp. *pallasiana* (Lamb.) Holmboe), Scots pine (*Pinus sylvestris* L.), and Taurus cedar (*Cedrus libani* A. Rich.) were collected from the Yukarıgökdere and Gölcük forest areas within the borders of Isparta, Türkiye, at different ages and altitudes (700-1700 m). The collected cone samples were cleaned of foreign matter and ground to a particle size of less than 0.5 mm. The samples were then homogenized and dried in an oven until a constant weight was reached. The prepared samples were carefully stored in glass containers and kept refrigerated until the start of the experimental procedures. Figure 1 shows the study area in the Isparta region, while Figure 2 shows conifer cones after seed production in local forest station.

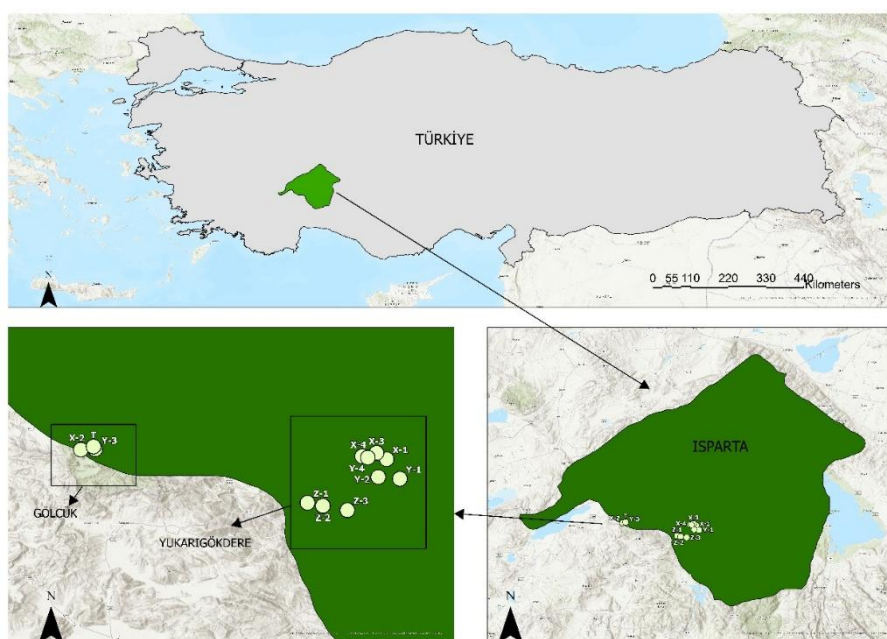


Figure 1. General map of study area



Figure 2. The conifer cones after seed production in local forest station (Isparta, Türkiye)

Calorific value measurements were performed using a bomb calorimeter calibrated with benzoic acid (LECO AC-500, St. Joseph, MI, USA). However, calorific value determination of the cone samples was carried out by weighing approximately 1 g of each sample on a precision balance, placing them in capsules, and igniting them using the device's automatic ignition system, in accordance with the American Society for Testing and Materials (ASTM) D5865 standard.

Total sulphur content was determined using the sulphur module option of the Epsilon EDRX Element Analyzer (PANalytical, Almelo, Netherlands) which detects SO₂ gas released during combustion carried out in an oxygen atmosphere. The ash content of the samples was determined according to the ASTM E1755 standard. Approximately 1-2 g of sample was carbonized in crucibles at 250 ± 10 °C and then burned in an ash furnace at 575 ± 25 °C until a constant weight was reached. After cooling the samples in a desiccator, the ash content (%) was calculated from the mass difference before and after combustion using the following equation (1).

$$\text{Ash (\%)} = \left(\frac{m_t}{m_0} \right) \times 100 \quad (1)$$

In the equation given above, m_0 represents the initial sample quantity, and m_t represents the amount of ash remaining after the combustion process.

The elemental composition of the ash samples (Ca, K, Mg, Fe, Si, Al, and other elements) was determined on pellets prepared from ash samples obtained from the furnace, using an Epsilon EDRX Elemental instrument (PANalytical, Almelo, Netherlands) in accordance with instrument standards. Analyses (calorific value, volatile matter, ash, and sulphur content) were performed in ten replicates according to relevant standards, and average values are presented in the tables. To determine functional groups, FT-IR analyses were performed using a JASCO FT/IR-4700 typeA instrument operating in the 400-4000 cm⁻¹ wavelength range, with approximately 1 cm⁻¹ resolution and in transmittance mode. The milled forms of the samples are shown in Figure 3.

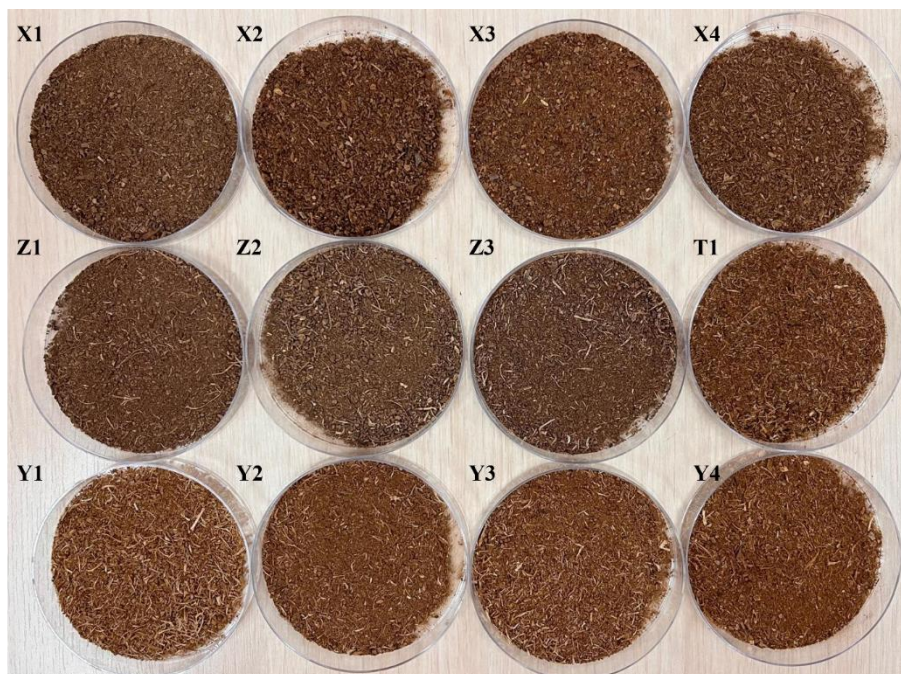


Figure 3. Milled forms of the samples (X: Taurus Cedar, Z: Turkish Red Pine, T: Scots Pine, Y: Black Pine)

3. Results and discussions

Table 1 shows the average calorie (fuel) values of the species at different elevations. The average calorific values (kcal/kg) measured at elevations between 700 and 1700 meters for four different coniferous species, namely Taurus cedar, Turkish red pine, black pine, and Scots pine, were compared. In Taurus cedar samples, it was observed that calorific values remained fairly constant between 4869 and 4846 kcal/kg in the 1300-1700 m elevation range and were not significantly affected by altitude. In Turkish red pine species, the value of 4796 kcal/kg at 700 m decreased to 4110 kcal/kg at 1000 m and increased to 4850 kcal/kg at 1300 m, suggesting the influence of environmental conditions or instinct differences. In black pine species, the calorific value decreased from 4914 kcal/kg to 4302 kcal/kg as altitude increased; this may be related to changes in wood chemical constituents beside density or moisture content at high altitudes. The only value measured for Scots pine at 1500 m is 4418 kcal/kg, which shows a medium energy potential compared to other species. Overall, the highest average calorific value among the species was found in Taurus cedar, and the lowest in black pine, highlighting the importance of species selection for biofuel or energy production.

Table 2 shows the calorific values, volatile matter, and sulphur content for different species code as (X, Y and Z groups). The calorific values were found to be in range from 3954 to 5050 kcal/kg, indicating the similar energy potential compare to wood. In samples belonging to the X group, calorific values were found to vary between 4668-5050 kcal/kg, indicating a high energy content. Calorific values in samples from group Z were found to range from 3954 to 5015

kcal/kg and were generally higher than those in group Y. However, calorific values in samples from group Y ranged from 4037 to 4487 kcal/kg, which is lower than the other groups, indicating that this type is less efficient in terms of energy compared to the others.

Volatile organic matter ratios vary between 58% and 64.24% across species. The lowest volatile organic matter ratio was observed in sample X3 (58%), while the highest ratio was detected in sample Z1 (64.24%). This difference can be attributed to the chemical composition and cellular structure of the cone. In general, high volatile organic matter ratios indicate that the cone is more flammable and can release energy more quickly during combustion. It has reported by researchers that there have diversity of volatile matters from forest biomass for both quantitatively and qualitatively, that direct impact on the energy properties of given substrates (Baker, 1983; White and Diertenberger, 2001).

Sulphur ratios were found to be quite low (0.02-0.10%). This indicates that all samples have environmentally clean fuel characteristics. The lowest sulphur content was observed in samples Y2 and Y3 (0.02%), while the highest was found in samples X3 and Z2 (0.10%). These findings reveal that cedar and red pine species, in particular, can stand out as environmentally friendly alternatives for biofuel due to their high calorific values and low sulphur content. However, forest biomass has generally contain very low amount of sulphur element in their structure, resulting environmentally friendly fuel sources (García et al., 2012). The result found in our study clearly support this literature informations.

Table 1. Average calorie values of species based on elevation

Code	Species	Elevation (m)	Calorific (kcal/kg)
X1	Taurus Cedar	1300	4869 ± 90.70
X2	Taurus Cedar	1500	4834 ± 94.77
X3	Taurus Cedar	1700	4846 ± 96.92
X4	Taurus Cedar	1400	4479 ± 92.09
Z1	Turkish Red Pine	700	4796 ± 98.60
Z2	Turkish Red Pine	1000	4110 ± 95.98
Z3	Turkish Red Pine	1300	4850 ± 94.92
Y1	Black Pine	1000	4353 ± 94.22
Y2	Black Pine	1300	4194 ± 93.87
Y3	Black Pine	1700	4302 ± 93.45
Y4	Black Pine	1400	4390 ± 95.42
T1	Scots Pine	1500	4418 ± 93.27

Table 2. General properties of species at various elevations and of similar age

Species	Calorie (kcal/kg)	Volatile matter (%)	Sulphur (%)
X1	4701-5036	62.19 ± 0.24	0.08 ± 0.02
X2	4668-5000	62.40 ± 0.26	0.07 ± 0.02
X3	4686-5005	58.00 ± 0.37	0.10 ± 0.03
Z1	4631-4961	64.24 ± 0.23	0.07 ± 0.02
Z2	3954-4265	64.04 ± 0.29	0.10 ± 0.03
Z3	4684-5015	63.20 ± 0.27	0.06 ± 0.02
Y1	4219-4487	62.30 ± 0.25	0.06 ± 0.02
Y2	4037-4350	64.00 ± 0.31	0.02 ± 0.01
Y3	4149-4455	62.17 ± 0.27	0.02 ± 0.01

In many studies in the literature, sulphur release has been used as an important criterion for evaluating the environmentally friendly fuel properties of an energy source. Consistent with these studies, fuels with the lowest sulphur emissions into the atmosphere have been reported to be more environmentally friendly (Demirbaş, 2004; Omer, 2017; Jomantas et al., 2025). Different biomass types may exhibit variation not only in their calorific values but also in their ash content (%) and the composition of their ash constituents, even when these components show considerable differences within the same species (Sahin, 2021; Sahin et al., 2023). Figure 4 compares the ash (%) and sulphur (%) values of four different species samples at the same elevation (1350 ± 50 m) and age group (73 ± 3 years). As seen in the graph, ash ratios vary between 1.2% and 1.7%. The highest ash ratio was detected in the X4 sample (1.7%), while the lowest was in the Y4 sample (1.2%). This indicates that the inorganic mineral content of the X4 sample is higher than that of the other species. Sulphur values are quite low, ranging from 0.02% to 0.09%. The highest sulphur content was observed in the T1 sample (0.09%), while the lowest value was determined in the Y4 sample (0.02%). In general, the low sulphur content in all samples indicates environmentally suitable properties for use as fuel. Furthermore, the limited ash content indicates high combustion efficiency and the suitability of the selected cone species for energy purposes.

Figure 5 compares the volatile organic matter (%) values of four different species samples with similar elevation (1350 ± 50 m) and age group (73 ± 3 years). When examining the graph, it can be seen that the volatile organic matter ratios show a decreasing trend among the species. The highest volatile matter ratio was detected in the X4 sample (66%), followed by Z3 (63.2%), Y4 (63%), and T1 (61.8%). A high volatile organic matter ratio indicates that the cone is more flammable and can form a gas phase more quickly during combustion. In this context, the X4 sample stands out as the species with the highest potential in terms of combustion reactivity. The lower volatile organic matter content observed in the T1 sample suggests that the combustion process for this

species may occur more slowly and that the rate of energy release may be relatively low. Overall, the fact that the volatile matter content in all samples is above 60% indicates that these woods possess suitable thermochemical properties for energy production.

There have been some studies related to energy properties of forest biomass. In a more recent study, Sahin (2021) investigated energy properties of three different above ground biomass components (branch, crown, bark) of European beech (*Fagus sylvatica*). He suggested that selected European beech's age groups have influenced combustion and calorific properties in some level while the highest volatile matter content of 84.6% was obtained with a 30 years old branch wood sample. However, it was also reported that the energy levels look like very similar in wood from crown- and branch parts but considerably lower for bark (3776.6 kcal/kg to 4200.7 kcal/kg) (Sahin, 2021). In our study, the volatile matter had changes as sample types changes. It could be expected considering heterogeneous constituents of wood species.

Figure 6 compares the average ash (%) values obtained from samples collected from different elevations but from a similar age group (90 ± 20 years). Upon examination of the graph, it is seen that ash ratios show significant differences between species. The highest ash ratio was determined in the X3 sample (2.1%), indicating that this species or elevation is richer in mineral accumulation than the others. Relatively high ash ratios (1.5-1.7%) were also detected in the X1 and X2 samples. In the Z group, ash content ranged from 0.9% to 1.7%, with the Z2 sample having the highest value within this group. In the Y group samples, ash content was generally lower; in particular, values were around 0.6% in the Y2 and Y3 samples. These results indicate that elevation and species characteristics affect ash content. In general, species with low ash content are more advantageous for energy production because ash reduces combustion efficiency and increases residue formation in the system. In this context, the Y2 and Y3 samples appear more suitable for biofuel potential due to their low ash content.

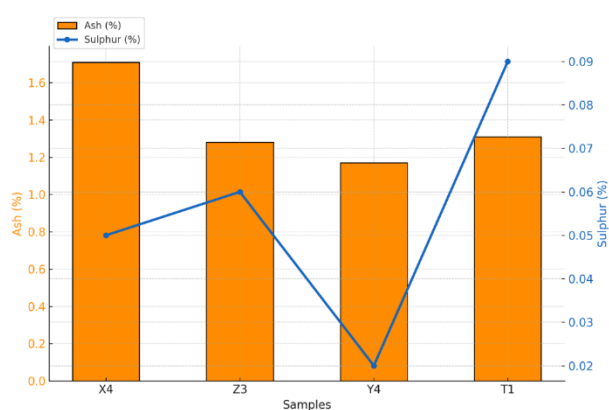


Figure 4. Ash (%) and sulphur (%) values of some species at various elevations and ages

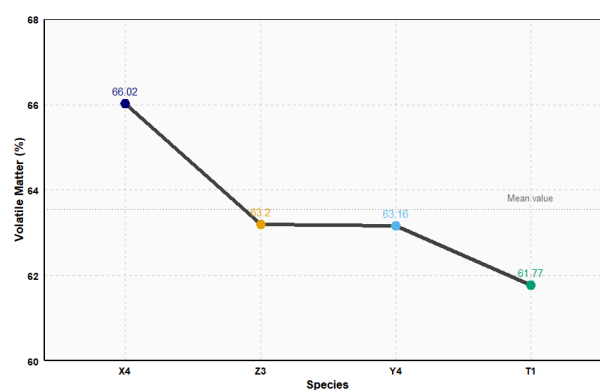


Figure 5. Volatile matter (%) values at similar ages and elevations

In general, it has proposed that the ash content of wood is around 0.6 to 2.0% and 3.0% or higher for barks (Young, 1992; Sahin, 2021). When Figure 5 is carefully evaluated, it appeared to there is no close relationship between ash content and altitude of samplings. Those could be expected considering forest biomass (trees) are non-homogenous combustion properties (Temesgen et al., 2015; Jekayinfa et al., 2020). However, Kumar et al. (2010) investigated effect of tree age on selected fuel properties of Eucalyptus hybrid species. They reported that the ash content Eucalyptus hybrids were between 0.4 and 1.1% and 0.8% for Eucalyptus nitens which those directly influenced for lowering calorific values in comparison with the wood fraction (Kumar et al., 2010). When considering obtained data in Table 2 and Figure 5, together, it is reasonably to suggest that there is a correlation between ash content and elevation of X samples but no correlation was obtained for Z and Y samples.

Figure 7 shows the average elemental compositions of different sample types. The graph compares the percentage distribution of SiO_2 , Al_2O_3 , Fe_2O_3 , CaO , MgO , K_2O , and P_2O_5 components. In general, SiO_2 and CaO components are found in higher proportions than other groups in all samples,

indicating that they represent the main inorganic components of the ash. The highest total element ratio was determined in the Z3 sample (approximately 3.1%), followed by the X3 and Y4 samples. Since these samples have a high mineral content, a high ash content is also expected. SiO_2 content is particularly dominant in the X3, Z3, and Y4 samples; this indicates that these types have a silica-rich structure and play an important role in ash formation. Al_2O_3 and Fe_2O_3 ratios were found at lower levels, generally varying between 0.1–0.3%. CaO and MgO components are important elements determining the basic character of the ash and were observed at high concentrations, especially in the Z2, Z3, and Y4 samples. Although the K_2O and P_2O_5 ratios are relatively low, they represent sources of potassium and phosphorus that could affect the melting behavior of the ash. Overall, the Z3 and X3 samples stand out with their high total element concentrations, while the Y2 and Z1 samples show the lowest element accumulation. These results reveal that species and elevation differences have significant effects on the chemical composition of ash and that silica and calcium content, in particular, are the key elements determining ash characteristics.

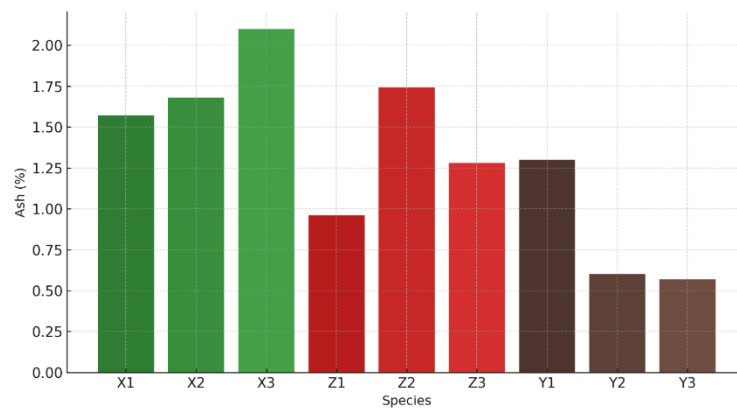


Figure 6. Average ash (%) values

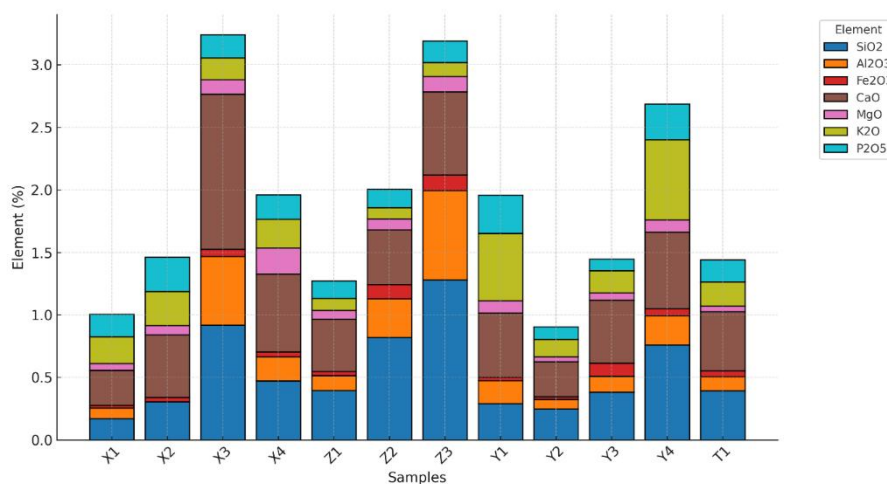


Figure 7. Average element composition (%) of species

Figure 8 shows the FT-IR spectra of cone samples obtained from different species. Fourier Transform Infrared Spectroscopy (FT-IR) enables the identification of characteristic functional groups by evaluating the molecular composition of materials through vibration spectra. The broad band in the 3400-3200 cm^{-1} region in all samples indicates hydroxyl groups (O-H stretching) found in cellulose, hemicellulose, and lignin. The bands in the 2920-2850 cm^{-1} range show aliphatic C-H stretching vibrations commonly seen in lignocellulosic structures (Rather and Mohammad, 2015; Belcaid et al., 2024).

The peak observed around $\sim 1735 \text{ cm}^{-1}$ corresponds to C=O stretching, representing acetyl and ester groups in hemicellulose. Vibrations in the 1600-1500 cm^{-1} range confirm the presence of aromatic C=C structures specific to lignin. The strong bands that become apparent in the 1250-1000 cm^{-1} region show C-O stretching vibrations belonging to alcohol, ether, and ester bonds found in hemicellulose and lignin, consistent with previous studies on polysaccharide structures (Nandgude et al., 2008; Rather and Mohammad, 2015; Bayram et al., 2024). The absorptions in the lower fingerprint region cover characteristic vibrations associated with C-H bends of aliphatic components and alkenes and alkyl halide structures (Swathilakshmi and Poonkothai, 2024; Özkan et al., 2025).

In this study, the effect of elevation levels (1-3) on calorific value (kcal/kg), volatile matter content (%), and sulphur content (%) was examined in samples belonging to

three different species (X, Y, and Z) at different elevations and similar ages. The values given in the tables indicate averages, and 10 repeated measurements were made for each species and elevation with a 5% error rate, and the data were statistically evaluated. In addition to the species and elevation factors, the statistical significance of the species x elevation interactions was evaluated using ANOVA and Tukey multiple comparison test. According to the findings, all three variables were affected by both species and elevation. The effect of species on calorific value is statistically significant ($p < 0.05$). Additionally, the effect of elevation was also found to be significant. According to the Tukey test, all elevation levels differ from each other. Furthermore, the species x elevation interaction effect is also significant, and it has been observed that different species respond differently to changes in elevation.

The species effect is also significant in volatile matter content. The volatile matter levels of the species are different ($p < 0.05$). The elevation factor was also found to be significantly effective. Although the sulphur content has low absolute values, the species effect is important. All species are statistically different from each other in terms of sulphur content ($p < 0.05$). Only in the altitude effect, no significant difference was found between the 2nd and 3rd levels ($p > 0.05$). Figure 9 shows the elevation-dependent regression graphs and the boxplot graphs for the species.

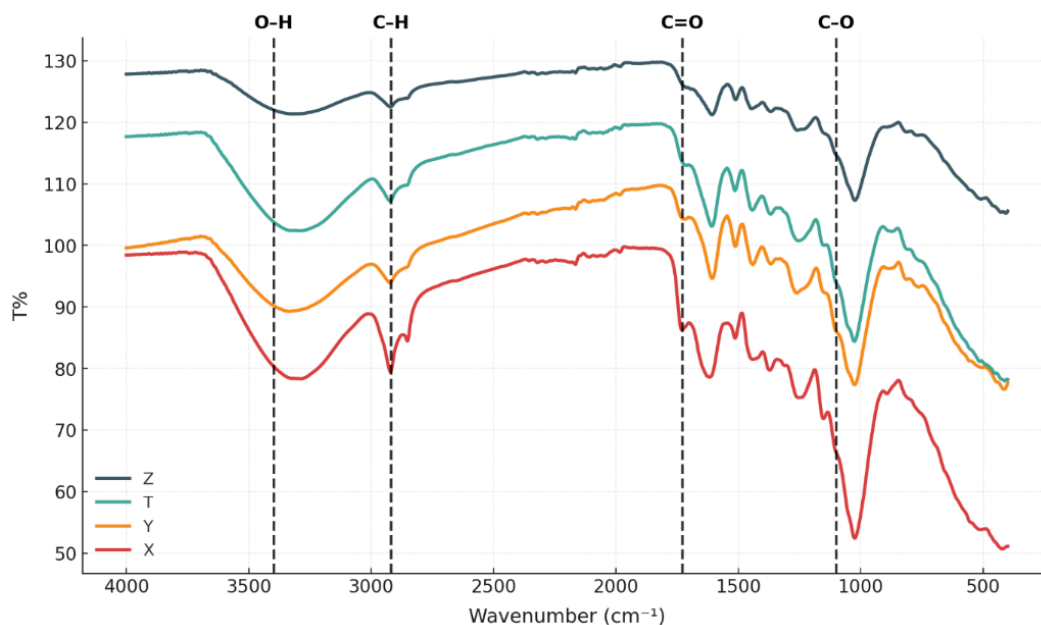


Figure 8. FT-IR spectra results

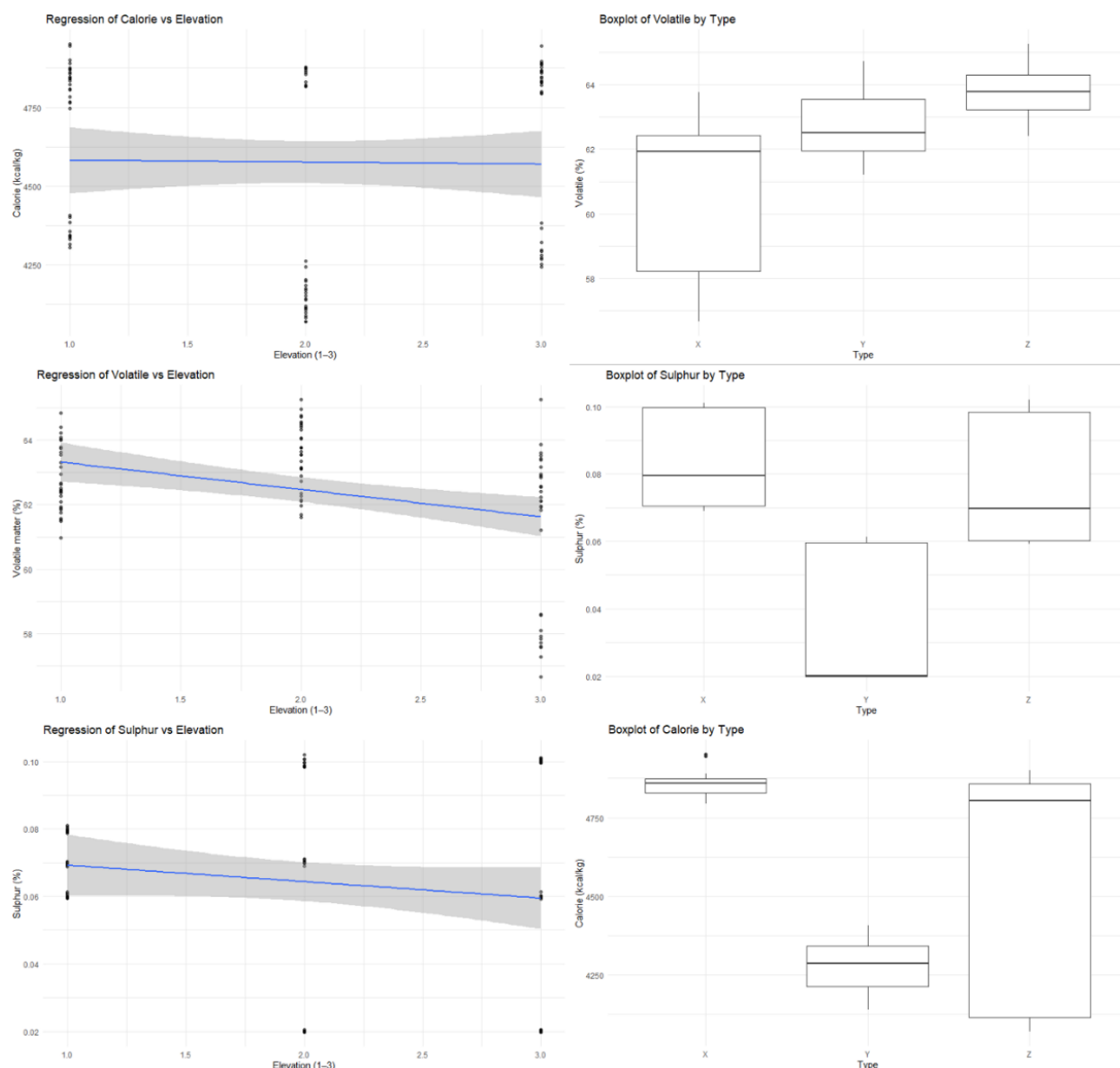


Figure 9. Elevation-dependent regression graphs and the boxplot graphs for the species

4. Conclusion

This study comprehensively evaluated the biofuel potential of cone residues from four coniferous species collected from different elevations in the Isparta region: Taurus cedar (*Cedrus libani* A. Rich.), Turkish red pine (*Pinus brutia* Ten.), Black pine (*Pinus nigra* subsp. *pallasiana* (Lamb.) Holmboe), and Scots pine (*Pinus sylvestris* L.) from the Isparta region. According to the analysis results, the calorific values of the cone samples ranged from 3954 to 5050 kcal/kg. The similarity of these values to traditional lignocellulosic fuels (e.g., wood) indicates that cones can be considered as an alternative biomass source. Among the species, Taurus cedar cones had the highest energy value, while black pine samples showed relatively lower energy content. This situation demonstrates the decisive effect of species differences and altitude variability on biofuel performance.

Volatile matter ratios (58-64%) and low sulphur contents (0.02-0.10%) indicate that pine cones are environmentally clean fuel materials with high combustion efficiency. Furthermore, low ash content and suitable elemental compositions rich in SiO_2 and CaO prove that these biomasses possess thermochemical properties suitable for clean energy production. FT-IR analyses revealed O-H, C-H, C=O, and C-O functional groups characteristic of lignocellulosic structures in all samples, confirming that the chemical structure of cone biomass is largely similar to that of wood.

When evaluated in terms of sustainability, the utilization of needle leaved cones, which become waste after seed extraction, in energy production can make significant contributions in terms of biomass recovery, increasing renewable energy diversity, and forest residue management. Thanks to their low sulphur emissions, high calorific value, and suitable combustion properties, Taurus cedar and red pine cones, in particular, stand out as promising raw materials for biofuel production.

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