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# REASSESSMENT OF RAPA WHELK SHELLS AND AN INNOVATIVE ROADMAP FOR INDUSTRIAL APPLICATIONS

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## Abstract

This study aims to determine the industrial and economic potential of Rapa whelk (*Rapana venosa*) shells as an economic product using characterization methods. Methods such as Zero Charge Point ($pH_{PZC}$), FT-IR analysis, scanning electron microscopy (SEM) images, EDS analysis, and X-ray diffraction analyses were employed. The results indicate that the $pH_{PZC}$ of Rapa whelk shells is 8.35, suggesting their surfaces will be electrically neutral at pH 8.35. X-ray diffraction analyses revealed that the main building material of the shells is aragonite-structured calcium carbonate ($CaCO_3$). SEM and EDS analyses demonstrated that the main components of the shells are carbon (C), calcium (Ca), and oxygen (O), with low levels of protein. FT-IR analyses confirmed the presence of $CO_3^{-2}$ vibration bands at 1407 cm-1, indicating that the shells are primarily composed of $CaCO_3$. These characterization methods provide essential information for evaluating the potential of Rapa whelk shells as an economic product. This study will significantly contribute to the sustainable utilization and industrial valorization of marine resources by understanding material's surface chemistry and interaction mechanisms.

**Keywords:** Rapa whelk, shells, aragonite, FT-IR, SEM, x-ray, $pH_{PZC}$

## Introduction

Today, seafood consumption is on the rise globally, driven by population growth and evolving dietary preferences (Bayraklı, 2021a,b; Bayrakli & Duyar, 2021; Duyar et al., 2023; Yıldız et al., 2023). However, this surge results in the generation of substantial amounts of waste in the seafood industry (Bayraklı & Duyar, 2019; Bayraklı, 2023 a, b; Duyar & Bayraklı, 2023). It is crucial to explore sustainable and effective methods for utilizing these processed seafood residues, as this holds significant environmental and economic importance. Failure to utilize seafood residues can result in environmental pollution and resource wastage (Wilson et al., 2009; Yang et al., 2019). Conversely, repurposing these residues not only reduces waste but also extracts valuable components. These extracted ingredients have versatile applications across industries including food, pharmaceuticals, cosmetics, and construction materials. One such component is the shells of Rapa whelks (*Rapana venosa*) (Eryaşar et al., 2018; Dağtekin 2023; Bayraklı et al., 2016). The structure of Rapa whelk shells is notable for its complexity, formed through biomineralization processes. Traditionally, these shells have been used for aesthetic or ornamental purposes. However, recent research has explored different applications to enhance the industrial and economic value of the shells. Particularly, the potential for utilizing the shells in various industrial applications holds significant importance in augmenting their economic value (Ji et al., 2015). It is crucial to note that Rapa whelks are an invasive species, especially known for their predation on mussel beds. As a result, their harvesting is actively conducted and encouraged to mitigate their impact on native ecosystems and promote biodiversity (Skein et al., 2020; Bayrakli, 2024). The Zero Charge Point ($pH_{PZC}$) is considered a critical parameter in controlling the surface charging of a material. Determining this parameter is important to understand the surface chemistry of the material and evaluate interaction mechanisms (Egbedina et al., 2021). FT-IR analyses are widely used spectroscopic techniques to determine the chemical composition and structural properties of a material. The FT-IR analysis of Rapa whelk shells enables the evaluation of the material's molecular composition and suitability for potential industrial applications (Tuo et al., 2010). Scanning Electron Microscopy (SEM) images and EDS analyses are important characterization techniques used to examine the morphology and surface properties of the shells in detail. These analyses assist in evaluating the microstructures, porosity levels, and chemical compositions of the shells (Parveen et al., 2020). X-ray diffraction analyses are used to determine the crystalline structures and mineral components of the shells. These analyses aid in gaining a deeper understanding of the material's mineralogical properties (Ji et al., 2013). The aim of this study is to determine and understand the potential of Rapa whelk shells as an economic product using a series of characterization methods. These methods include determining the Zero Charge Point ($pH_{PZC}$), FT-IR analyses, scanning electron microscopy (SEM) images and EDS analyses, X-ray diffraction analyses.

This article will present the results of these characterization studies conducted to assess Rapa whelk shells as an economic product and discuss their potential in various industrial applications. This study will contribute significantly to the sustainable utilization and industrial valorization of marine resources.

## Material and Method

### *Sample collection*

The Rapa whelk samples were collected from the Yakakent region of Samsun between December 2014 and November 2015. Fishing for Rapa whelks with the algarna is prohibited in the Black Sea between April 15 and August 31. In this study, special permission was obtained from the ministry for the prohibited periods.

### *Determination of Zero Load Point of Shells ($pH_{PZC}$)*

The pH-dependent zero charge points (PZC) of all obtained and ground shell products were determined. For the determination of $pH_{PZC}$, 0.01 M KNO3 solutions were first prepared in a 100 mL flask. The initial pH of these solutions (pHB) was adjusted between pH 4 and 10 with 0.1 M HCl and NaOH. The modified samples were then added to these solutions. The solution was then stirred with a magnetic stirrer for 48 hours at constant temperature. After 48 hours, the final pH of the solution (pHs) was measured and recorded. The difference between initial pH and final pH (ΔpH= pHB - pHs) was plotted against the initial pH (pHB). The point where the curve crosses the x-axis was determined as PZC.

### *FT-IR Analysis*
The FT-IR spectra of the shells were measured in the range of 650-4000 cm-1 with the ATR technique on the Perkin-Elmer SpectrumOne device at Çanakkale Onsekiz Mart University, Central Laboratory.

### *Scanning Electron Microscopy (SEM) Image and EDS Analysis*
SEM image and Energy Dispersive X-Ray spectroscopy (EDS) analysis were performed with Jeol (JXA 8230) brand device using carbon coating method. The magnification capacity of the device is between ×40 and 300.000 and the accelerator voltage is between 0.2 and 30 kV. EDS analysis of all samples obtained within the scope of the study was also performed. Images were taken using secondary electrons in the SEM device. Since the surfaces of the materials were coated with carbon, carbon was not evaluated in the EDS results.

### *X-Ray Diffraction Analyses*
The X-Ray Diffraction method (XRD) is based on the principle that each crystalline phase diffracts X-rays in a characteristic pattern depending on their unique atomic arrangement. The shell of the sample was ground into powder with a mortar and pestle and then analyzed with the PANalytical Empyrean brand X-RD device in the Central Laboratory of Canakkale Onsekiz Mart University (Çanakkale - Türkiye).

## Results and Discussion
### *Determining the Zero Charge Point*
The PZC is a critical parameter that defines the pH at which a material surface is electrically neutral. At this point, the material exhibits no net electrical charge, leading to equal attraction and repulsion of positively and negatively charged ions. The PZC value of 8.35 for Rapa whelk shell particles indicates that their surface will be neutral at a pH of 8.35 (Figure 1). Below the PZC (pH < 8.35), the surface of the Rapa whelk shell particles will attract positively charged ions while repelling negatively charged ions, resulting in a positive electrical charge on the surface. Conversely, above the PZC (pH > 8.35), the particles will attract negatively charged ions and repel positively charged ions, leading to a negative electrical charge on the surface. Understanding the PZC value is crucial for predicting and controlling the surface properties of particles. The surface charge influences various processes such as dispersion stability, adsorption, and precipitation. For instance, the surface charge of particles can impact their interactions with other substances, affecting their stability in suspensions, their ability to adsorb onto surfaces, and their tendency to form precipitates (Kim et al., 2021).

Overall, the provided information on the PZC of Rapa whelk shell particles and its implications on surface charge and interactions is scientifically sound and aligns with the fundamental principles of colloid and surface chemistry (Singh et al., 2015).

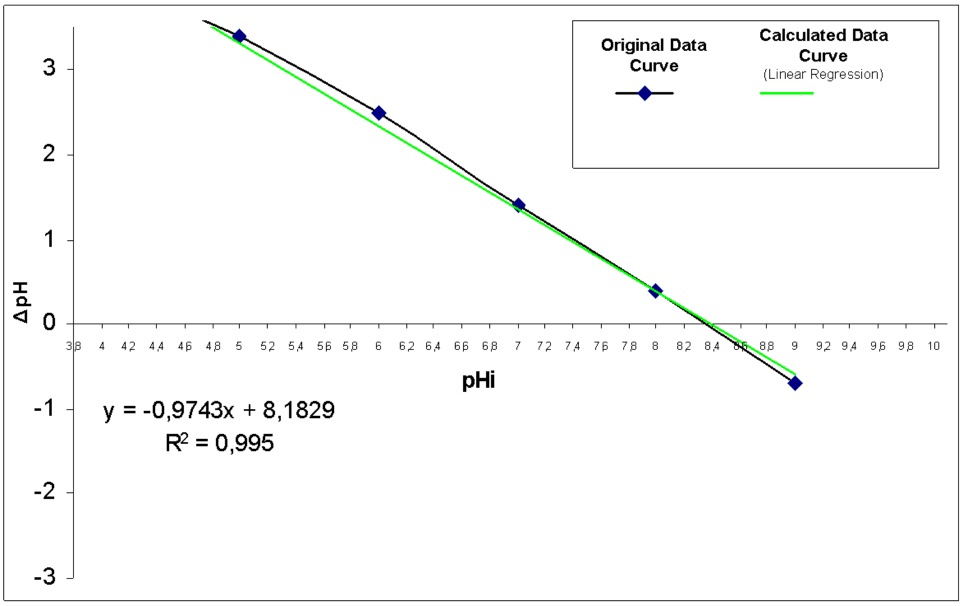


**Figure 1**. Determination of Zero Charge Point Value for Rapa whelk Shell Particles: Curves of Initial pH (pHi) versus ΔpH

***X-Ray Diffraction (X-RD) Results of the Rapa Whelk Shell***

In this research, Rapa whelk shells were analyzed by the X-Ray Diffraction method (X-RD) and the data were compared with calcium carbonate and aragonite ($CaCO_3$) crystal structures. Analyzes are based on examining the atomic and molecular structure of a material and refracting X-rays in a characteristic pattern depending on the crystal phase of the structure. Diffraction properties can identify that crystal like a fingerprint. According to the findings of this study, it was revealed that the main building material of the Rapa whelk shell is calcium carbonate ($CaCO_3$). However, the crystal structure of $CaCO_3$ in the shell is different from the calcite structure used as a reference. X-RD results show that the $CaCO_3$ in the Rapa whelk shell has an aragonite crystal structure. Aragonite is a carbonate mineral with the same chemical composition as calcite but with a different crystal lattice structure. Aragonite is typically biologically formed in marine or fresh waters. For this reason, the crystal structures were analyzed to determine which structure is suitable for the shell structure of the species (Rajan et al., 2023; Koga et al., 2013). Figure 2 shows the X-RD spectrum of a mixture of calcite (C) and aragonite (A) made by the U.S. Geological Survey. This spectrum is a suitable data for comparison of X-RD results of crusts. All spectra were compared with the data obtained from "The Database of the RRUFF" website. When the spectra in Figure 3 are examined, it is seen that aragonite, which is of biogenic origin, is far from the calcium carbonate crystal structure. In the spectra, the blue part represents samples, the red part represents calcium carbonate and the green part represents aragonite. The formation of the aragonite structure in the Rapa whelk shell is related to the shell's growth conditions and environmental factors (Ji et al., 2015; Duquette et al. 2017). Aragonite is more soluble and less stable than calcite, so Rapa whelks can produce aragonite instead of calcite to form a harder and durable Shell (Li et al., 2009). X-RD analysis can be a valuable tool to compare the shell structures of different Rapa whelk species and understand the factors affecting shell formation.

Aragonite is an important mineral used in various industrial applications. Aragonite is used as an aggregate in the production of cement and concrete. It is also used as a plasticizer and opacifying agent in the production of glass and ceramics (Islam et al., 2013). In metallurgy, it is used to improve slag flow in steel production (Wang et al., 2021). In agriculture, Aragonite applications are used as a soil conditioner to raise the pH of the soil and provide calcium (Hussein et al., 2020). It is a semi-precious stone also used in the jewelry industry. Aragonite's unique crystal structure and properties make it valuable for these various industrial applications.

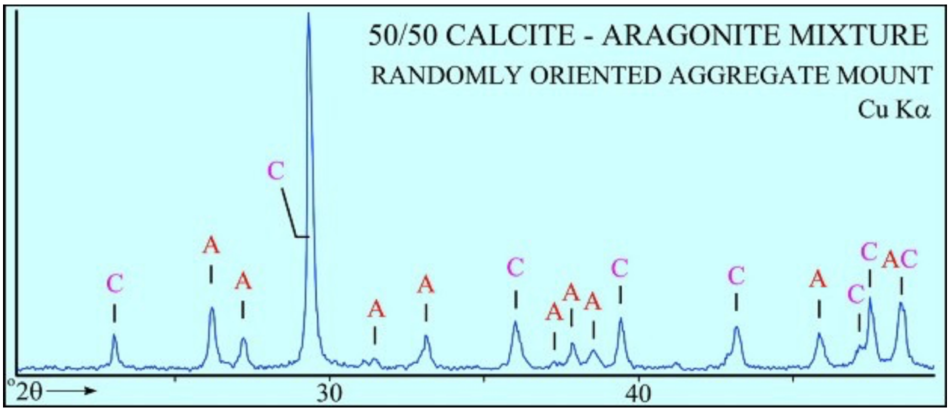


**Figure 2**. X-RD spectrum of a mixture of calcite and aragonite (US, 2001 geological survey results)

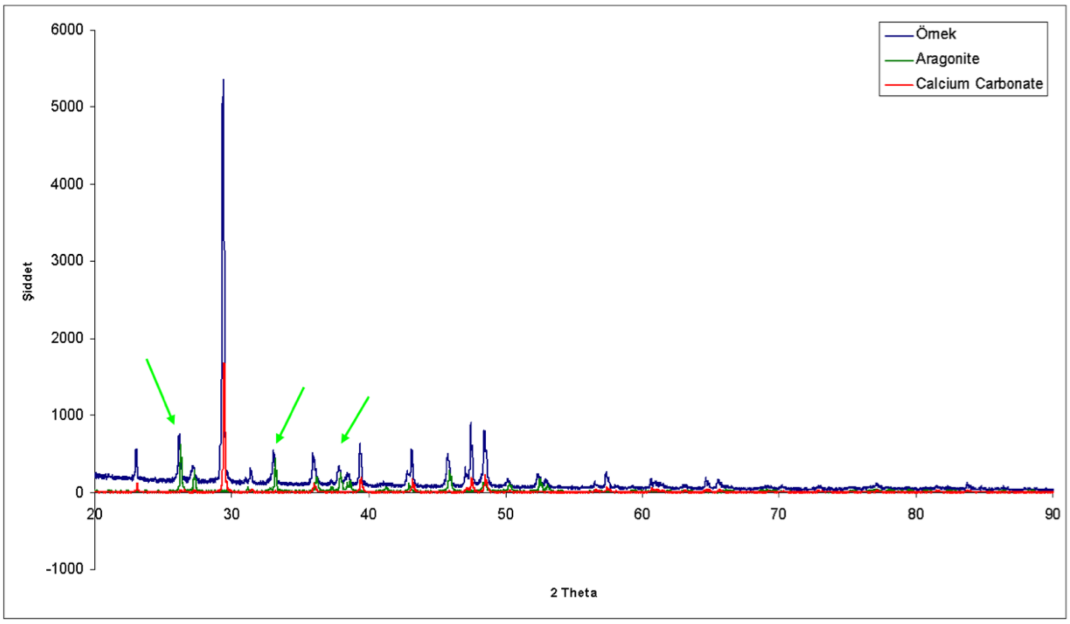


**Figure 3**. X-ray diffraction (X-RD) spectrum of Rapa whelk shell

***Scanning Electron Microscopy (SEM-EDS) Analysis Results***

SEM analysis of Rapa whelk shells and EDS analysis were performed to determine the elemental contents. SEM images of the shells are given in Figure 4. EDS analysis revealed that the main components of Rapa whelk shells are carbon (C), calcium (Ca) and oxygen (O). These elements form calcium carbonate ($CaCO_3$), the main building material of the shells. Furthermore, EDS analysis detected low levels of protein in the shells. Nitrogen (N) element (5.0%) belonging to proteins is also seen in the analysis results. According to the EDS results, the $CaCO_3$ component has the highest proportion of oxygen (55.8%). The amount of carbon (28.3%) is higher than calcium (10.2%), indicating that carbon comes from both $CaCO_3$ and protein.

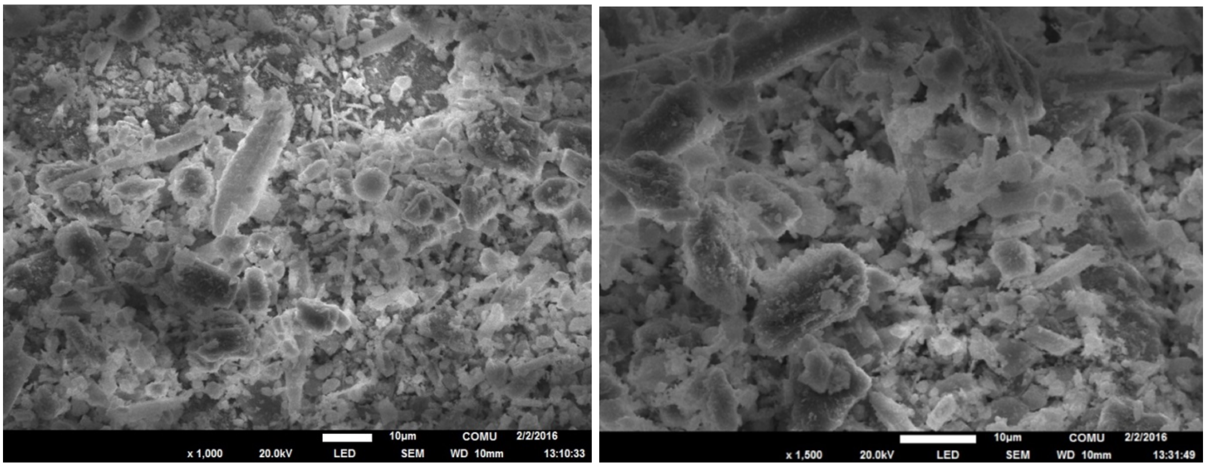

**Figure 4**. SEM images of Rapa whelk shells

EDS analysis results confirm that Rapa whelk shells are mainly composed of $CaCO_3$. The crystal structure of $CaCO_3$ provides the hardness and durability of the Shell (Zou et al., 2019). These results are in line with other studies in the literature and make an important contribution to identifying the major components of Rapa whelk shells. The protein detected in the shells is part of the organic matrix of the shell and helps to maintain the shape and structure of the Shell (Joubert et al., 2010; Liu et al., 2015). This finding highlights the importance of the balance between biological and mineral components of Rapa whelk shells. The low level (0.2%) of sodium (Na) detected in the EDS analysis supports the fact that Rapa whelks absorb calcium and carbonate ions from seawater and use them to build their shells. These results suggest that environmental factors play an important role in the shell-forming process of Rapa whelks. This study demonstrates that the chemical composition of Rapa whelk shells is influenced by species structure and environmental factors. Research on differences in the chemical composition of Rapa whelk shells between species and the effects of environmental conditions (Rajan et al., 2023; Parveen et al., 2020; Eryaşar et al., 2018; Ji et al., 2015; Ji et al., 2013) can help us understand shell formation and identify changes in shell composition. EDS analysis can be a valuable tool to compare the shell compositions of different Rapa whelk species and understand the factors affecting shell formation. Future studies can increase the knowledge in this field by investigating the biomineralization processes of Rapa whelk shells in more depth.

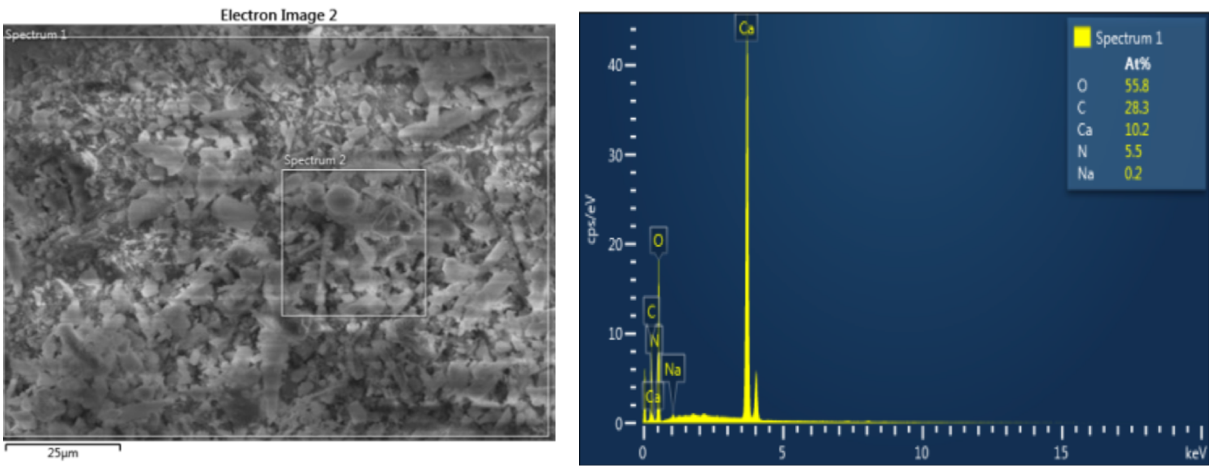

**Figure 5**. EDS spectra of Rapa whelk shells

***Infrared Spectroscopy (FT-IR) Analysis Results***

Infrared Spectroscopy (FT-IR) spectra of Rapa whelk shells are presented in Figure 6. Upon examination of the FT-IR data of the shell, vibration bands of $CO_3^{-2}$ at 1407 $cm^{-1}$ were observed. This band distinctly indicates the presence of carbonate, which constitutes the main chemical structure of the shells. The FT-IR results confirm that Rapa whelk shells are primarily composed of $CaCO_3$. The crystal structure of $CO_3^{-2}$ provides the shells with hardness and durability. The vibration band of $CO_3^{-2}$ at 1407 $cm^{-1}$ is a characteristic feature observed in the FT-IR spectra. This band corresponds to the asymmetric stretching vibration of the carbonate ion ($CO_3^{-2}$). This study demonstrates the valuable utility of FT-IR spectroscopy in characterizing the chemical composition of Rapa whelk shells. FT-IR analysis can be employed to compare shell compositions among different Rapa whelk species and understand the factors influencing shell formation (Ji et al., 2013). Additionally, FT-IR analysis can provide insights into the organic components of the shells. For instance, proteins present in the shells exhibit characteristic amid I bands in the region of 1650-1630 cm-1. These bands can be utilized to understand the structural properties of proteins and their roles within the shell. Future studies may integrate FT-IR spectroscopy with other analytical techniques to further investigate the chemical composition and structure of Rapa whelk shells in detail. Such studies will contribute to a better understanding of the mechanisms of shell formation and the adaptation of Rapa whelks to environmental conditions.


**Figure 6**. FT-IR spectrum of the shell

**Conclusion**

In conclusion, the comprehensive analysis of Rapa whelk shells using FT-IR spectroscopy, EDS, SEM, and X-ray diffraction has provided valuable insights into the chemical composition and structure of the shells. The combination of these techniques confirmed that the shells are primarily composed of calcium carbonate ($CaCO_3$) and contain organic components such as protein. EDS analysis revealed the presence of various elements like calcium, carbon, oxygen, and magnesium in the shells, while SEM images displayed a complex and ordered microstructure. X-ray diffraction further confirmed the aragonite crystal structure of the shells. The findings underscore the significance of FT-IR spectroscopy, EDS, SEM, and X-ray diffraction as essential tools for characterizing Rapa whelk shells. These techniques can be effectively utilized to compare shell compositions among different rapa whelk species, understand the factors influencing shell formation, and explore potential industrial applications of the shells. To further advance the understanding of Rapa whelk shells and their industrial applications, future studies could consider integrating FT-IR spectroscopy, EDS, SEM, and X-ray diffraction to compare shell compositions across Rapa whelk species. Additionally, combining these analytical techniques with investigations into the organic components of the shells and the impact of environmental factors on shell development could provide deeper insights into shell formation mechanisms and potential industrial uses. The integration of advanced analytical techniques and interdisciplinary approaches will not only enhance our knowledge of Rapa whelk shells but also pave the way for sustainable utilization and innovative applications of these natural resources.

**Ethical approval**
Not applicable

**Informed consent**
Not available

**Data availability statement**
The authors declare that data can be provided by corresponding author upon reasonable request.

**Conflicts of interest**
There is no conflict of interests for publishing this study.

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**Contribution of authors**
Barış Bayraklı : Funding acquisition, Conceptualization, Methodology, Data curation, Writing original draft, Software, Editing
Harun Yıldız : Investigation, Methodology, Writing original draft, Software, Editing
Serdar Baktaş : Investigation, Methodology, Writing original draft, Software, Editing
Bayram Kızılkaya : Investigation, Methodology, Writing original draft, Software, Editing.

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