POLİTEKNİK DERGİSİ

JOURNAL of POLYTECHNIC

ISSN: 1302-0900 (PRINT), ISSN: 2147-9429 (ONLINE)

URL: http://dergipark.org.tr/politeknik

Investigating the impact of growth time and methane flow on graphene synthesis using nickel foil

Nikel folyo üzerinde büyüme süresi ve metan akışının grafen sentezi üzerindeki etkisinin incelenmesi

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Bu makaleye şu şekilde atıfta bulunabilirsiniz (To cite to this article): Bozkaya M. Altuntepe A., Ateş H. ve Zan R., "Nikel folyo üzerinde büyüme süresi ve metan akışının grafen sentezi üzerindeki etkisinin incelenmesi", **Politeknik Dergisi, 24(3): 1181-1186, (2021).**

Erişim linki (To link to this article): http://dergipark.org.tr/politeknik/archive

DOI: 10.2339/politeknik.869535

Investigating the Impact of Growth Time and Methane Flow on Graphene Synthesis using Nickel Foil

Highlights

- *Graphene*
- *CVD*
- *Nickel*
- *Growth time*
- *Raman Spectroscopy*

Graphical Abstract

Growth time and methane rate were optimized for graphene synthesis on nickel substrate by CVD method.

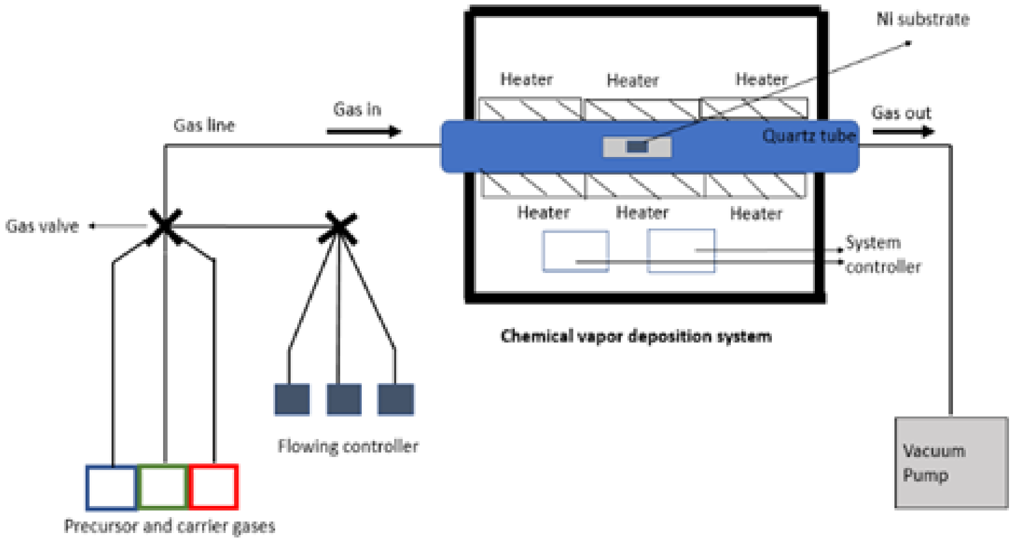

Figure. CVD system scheme

Aim

Growth time and methane flow rate parameters optimized in graphene film synthesis on nickel foil by the CVD method.

Design & Methodology

CVD method was used for homogeneous and large scale graphene synthesis to investigated the growth time and methane flow rate parameters of graphene synthesis on nickel foil.

Originality

Growth time and methane flow rate optimized for large-scale homogeneous graphene synthesis.

Findings

Single layer graphene was synthesized with a flow of 20 sccm methane.

Conclusion

Single layer homogeneous graphene was synthesised on nickel substrate.

Declaration of Ethical Standards

The authors of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

Nikel Folyo Üzerinde Büyüme Süresi ve Metan Akışının Grafen Sentezi Üzerindeki Etkisinin İncelenmesi

Araştırma Makalesi / Research Article

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(Geliş/Received : 28.01.2021 ; Kabul/Accepted :18.02.2021 ; Erken Görünüm/Early View : 01.03.2021)

ÖZ

Grafen, yüksek taşıyıcı mobilitesi, yüksek esneklik ve görünür bölgede yüksek ışık geçirgenliğinden dolayı en çok araştırılan nanomalzemelerden biri haline gelmiştir. Birçok üretim tekniği olmasına karşın kimyasal buhar biriktirme yöntemi, homojenlik, geniş ölçekli gafen sentezi gibi özellikleri sayesinde grafen üretiminde en çok kullanılan yaklaşım haline gelmiştir. Yüksek kalitede geniş ölçekli sentez için bu teknikte bir çok parametre etkili olmaktadır. Bu çalışmada, nikel folyo üzerinde büyütülen grafen için metan akış hızı ve büyüme süresi olmak üzere iki büyüme parametresi incelenmiştir. Farklı metan akış hızları (20 sccm'den 50 sccm) ve büyüme süresi (50 dakiakadan 20 dakikaya) ile büyütülen grafen filmler incelenmiştir. Tek katmanlı grafen filmin, Raman spektroskopi ölçümleriyle gösterildiği üzere, 20 dakika büyüme süresinde yalnızca 20 sccm metan akışı altında sentezlenebildiği belirlenmiştir. Ayrıca, tek katmanlı grafen filmin, Raman haritalaması ile teyit edildiği üzere yüksek homojenlikte olduğu belirlenmiştir. Öte yandan, hem metan akışını hem de büyüme süresini artırılarak çok katmanlı grafen filmi elde edilmiştir.

Anahtar Kelimeler: Grafen, CVD, nikel, büyütme, raman spektroskopisi.

Investigating the Impact of Growth Time and Methane Flow on Graphene Synthesis using Nickel Foil

ABSTRACT

Graphene has been one of the most investigated nanomaterials in recent years due to its one atom thickness nature and its extraordinary properties such as high conductivity and excellent transmittance in visible range, bendability and high carrier mobility. Although there are many techniques to grow graphene, chemical vapor deposition technique is one of the best approach to synthesize homogeneous and commercial scale graphene in thin film form. Many parameters can affect the quality and homogeneity of the graphene film that is grown with this technique. In this study, two growth parameters which are methane flow rate and growth time were investigated to find out their effect on the quality of the graphene film, which is grown on nickel foil. Graphene film grown with different flow rates of methane (from 20 to 50 sccm) and growth time (50 to 20 mins.) were examined. We found that single layer graphene film could only be grown under 20 sccm methane flow in 20 mins. growth time as evidenced via Raman spectroscopy measurements. Furthermore, the single layer graphene film was found to be in high homogeneity as confirmed by Raman mapping. On the other hand, multi-layer graphene film was obtained by increasing both methane flow and growth time.

Keywords: Graphene, CVD, nickel, growth, raman spectroscopy.

1. INTRODUCTION

Graphene consists of carbon atoms that are arranged in honeycomb structure and it is just one atom thick [1,2]. Since in 2004 when Geim and Novoselov seperated graphene flakes from graphite via scotch tape, which is called mechanical exfoliation, carbon based-nanomaterials have been the center of attention thanks to their superior properties. For example single layer graphene film has high optical transmittance in visible range (97%), high mobility (200 000 $cm^2/V.s$), ballistic transport, high flexibility and high strength [1]. Interestingly, such fascinating properties of graphene show diversity in accordance with the number of layers. Additionally, the properties of graphene depend not only on the number of layers but also on the method of synthesis. Single layer graphene has two conic points, K and K` in Brillouin zone which are named Dirac cones and electrons exhibit a linear dispersion near the Dirac point in the single layer graphene. However, electrons show parabolic electronic dispersion in double layer graphene and electrons behave massive Dirac fermions rather than Schrödinger equation unlike single layer graphene [3,4]. This special band structure makes single layer graphene an attractive material to be researched. Despite the missing band gap, graphene is still a very

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good candidate to be employed in many electronic applications thanks to its excellent properties such as high carrier mobility, stability and fractional quantum hall effect at the room temperature [5, 6].

Many techniques have been developed for graphene synthesis such as mechanical exfoliation, epitaxial growth on SiC, liquid-phase exfoliation and chemical vapor deposition (CVD) [7-9]. Mechanical exfoliation was reported as the first method to fabricate graphene films by exfoliation of graphite. Despite the relatively small size, the graphene film obtained with this approach is the best in terms of quality as their properties are close to the theoretical value [1]. On the other hand, the graphene synthesized epitaxially on SiC can be obtained in wafer-size; however, controlled sublimation of silicon is a challenge and requires high temperature (>1500 °C) [10]. At the same time, chemical and liquid phase exfoliation techniques are known to be the other successful approaches for industrial graphene fabrication due to their scalability and low cost. The electrical conductivity and carrier mobility of graphene obtained with these techniques are low in comparison to the graphene synthesized with the other techniques due to small size and oxygen content in the structure [11]. Thus, graphene obtained with these techniques are not much preferred for optoelectronic devices, which requires films with high homogeneity. Among those techniques, CVD is one of the mostly used one that enables high quality and large-scale graphene films [12,13].

Graphene, in thin film form, obtained using CVD method is grown on transition metal substrate such as Nickel (Ni), Cooper (Cu), Platinum (Pt), Iridium (Ir), and Gold (Au) by using various solid, liquid and gas precursors as carbon sources during the synthesis [14]. Methane (CH_4) is one of the popular carbon sources for the graphene synthesis because of its higher purity compared to the liquid and solid precursors [15]. In the CVD approach, there are many parameters such as temperature, pressure, growth and annealing time, flow rate of carbon sources [16,17] that can affect graphene quality. Further, another important parameter is the substrate, where the graphene film is grown on. In previous studies, researchers demonstrated that graphene synthesis could be accomplished on Cu and Ni substrates. However, there are two different growth mechanisms to synthesize graphene on these substrates [18]. Thanks to the low solubility of carbon in copper at high temperatures, single layer graphene synthesis on copper is known to be easier in comparison to the other metal catalyst as the growth on copper foil is mainly ruled by surface controlling. In the case of nickel substrate, where carbon solubility is quite high, firstly the decomposed carbon atoms diffuse into the substrate at high temperature and then carbon atoms precipitate during the cooling process, which is called precipitation and decomposition mechanism [19]. Thus, the graphene films can be grown easily on the nickel foil surface due to the successful decomposition of carbon at high temperature and low pressure [20]. However, controlling the graphene film thickness is quite challenging due to high solubility of carbon in the nickel foil. For this reason, preventing precipitation of extra carbon during graphene growth is important and it can be achieved by controlling many growth parameters [21]. For example, the flow rate of methane and growth time are critical parameters to control graphene film thickness on the nickel surface.

In this paper, the flow rate of methane and growth time were investigated to control both the growth of graphene film and its thickness on nickel foil using CVD approach. To do this, firstly, the flow rate of CH_4 was examined by changing it from 20 sccm to 80 sccm and the growth time was kept constant as 20 min. Then, the growth time was altered from 20 min. to 50 min. by setting the flow rate of CH_4 as 20 sccm. The synthesized graphene films were analyzed by Raman spectrometer, which was equipped with a 633 nm wavelength laser.

2. MATERIAL and METHOD

Graphene was grown on 25 µm polycrystalline Nickel foil using two-zone CVD system. Ahead of the synthesis process, the standard cleaning procedure was applied for Nickel foil. To do this, the foil was kept in acetone (5 min.), in deionized water (5 min.) and isopropyl alcohol (5 min.) during the pre-cleaning of the surface. The second step regarding pre-cleaning is to apply plasma cleaning to the foils in order to assure the elimination of most of the organic and inorganic contaminations. Then, the substrate was loaded to the CVD system, which was initially pumped down to $1x10^{-2}$ mbar. The schema of the typical CVD system is shown in Figure 1.

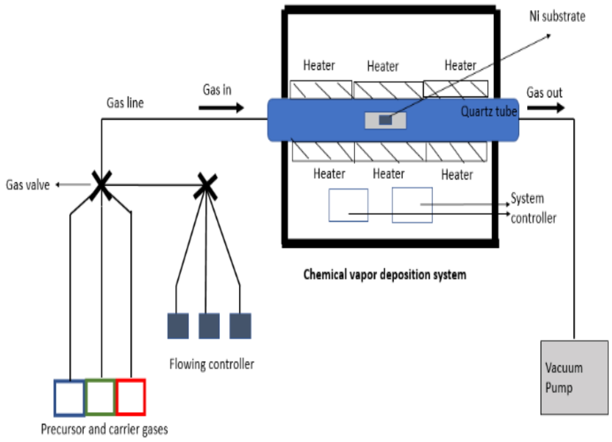

Figure 1. Schematic presentation of chemical vapor deposition system

During the growth processes, the CVD system was firstly heated up to 1000 °C in 60 min. at low pressure and then the foil was annealed for 30 min. at 1000 °C in order to increase the grain size in the foil, which is important for high quality graphene growth. The graphene growth was performed in 20 min. at 1000 °C and during the growth processes, 20 sccm Ar and 80 sccm H_2 gas flow was sent to the system. The growth time, annealing time, H_2 and

Ar flow rates were kept constant for all the experiments which were conducted in this research.

As mentioned above, the effect of methane flow and growth time were investigated in this study. So, as a first step in the study, different CH_4 flow rates (80/60/40/20 sccm) were applied to synthesize graphene films to find out the optimum methane flow regarding high quality graphene. After the determination of optimum methane flow, which was found to be 20 sccm, the graphene growth was further optimized via growth time amendment. To do this, the growth time was changed from 20 min. to 50 min. (20/30/40/50 min.) during the growth process. Additionally, H_2 (20 sccm), Ar (80 sccm) and CH_4 (20 sccm) gas flow rates along with 30 min. annealing time at 1000 ^oC were applied during the growth similar to the previous processes.

The graphene films were grown using different methane flow and different growth times which were characterized with Raman spectrometer using 633 nm wavelength laser excitation. The Raman peak positions, intensities and their ratios along with Raman mapping were employed for the determination of the film quality in terms of thickness and homogeneity.

3. RESULTS AND DISCUSSION

In the literature, Ma Prado Lavin-Lopez et. al. synthesized graphene on nickel substrate. Temperature effect was investigated in their study and they found that about 77% of the Ni foil was covered with monolayer graphene [22]. L. Huang et. al. optimized the growth time and H_2 flow rate for high quality graphene synthesis on the nickel substrate [23]. However, the growth time and CH_4 flow rate were not optimized for high quality and homogeneous single graphene synthesis. In the present study, the growth time and CH_4 flow were optimized in order to synthesize high quality graphene films. Raman spectroscopy is one of the essential characterization methods to analyze the structure of carbon-based materials such as graphene. There are three prominent peaks in graphene Raman spectrum, which are D, G, and 2D peaks. The D peak appears in the case of defects, impurities and contamination in the graphene structure. That peak emerges at about the 1350 cm^{-1} in the Raman spectrum. The G peak is placed around 1582 cm^{-1} band position and this peak represents sp^2 hybridization of carbon bonding. The 2D peak shows up in the 2700 cm^{-1} band position that is known as an indicator for the presence of the single layer graphene [22-24]. These peak positions, I_{2D}/I_G ratio and full width half maximum (FWHM) 2D peak are used to determine the quality of the graphene films [25-27].

The Raman spectra of the graphene films synthesized on the nickel foil using different CH_4 flow ratios are given in Figure 2. Based on these spectra of graphene films, few layer graphene film was obtained with 80 sccm methane flow during the growth and presence of the D peak confirmed surface contamination and/or defects. However, when the flow rate of methane gas was lowered to 60 sccm, the 2D peak was observed to be sharper than the previous sample at Raman spectrum. Moreover, we observed a significant decrease in D peak intensity, which is a good sign towards high quality graphene. The film quality was further increased by reducing the methane flow to 40 sccm in a better quality than the previous ones in terms of thickness and homogeneity.

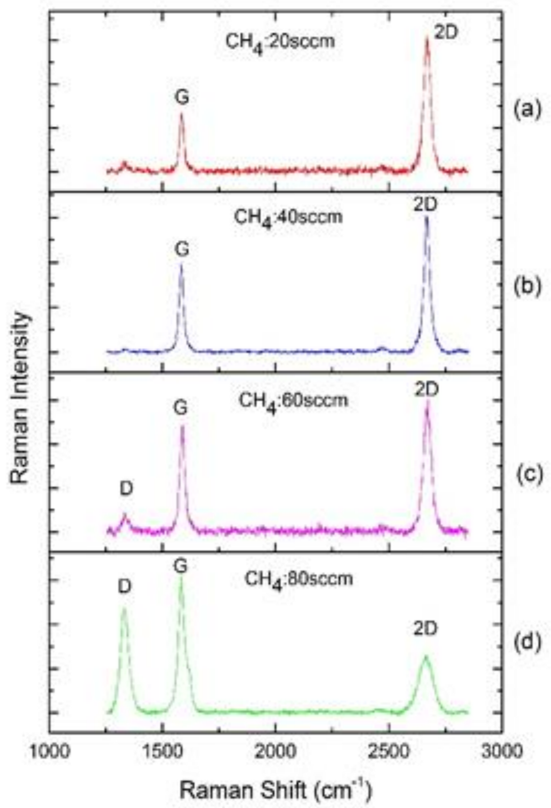

Figure 2. Raman spectra of the graphene films under the various flow of methane (a-d) 80-60-40-20 sccm, respectively

The film thickness was evaluated to be a double layer using I_{2D}/I_G ratio and 2D peak FWHM values. Moreover, the D peak was not observed on this film spectrum. Finally, single layer graphene was obtained by decreasing the flow rate of methane to 20 sccm. The details of those spectra in terms of I_{2D}/I_G ratio and 2D peak FWHM values are provided in Table 1. The I_{2D}/I_G ratio of the film was found to be 2.56, 1.55, 0.64, and 0.42 for the films grown with 20 sccm, 40 sccm, 60 sccm, 80 sccm CH_4 flow rates respectively. The I_{2D}/I_G ratios are decreased with the increase of the CH_4 flow rate, which supports our claims regarding the worsening film quality. In addition to the increase in the I_{2D}/I_G ratios, the decrease in the 2D peak FWHM value further confirms the increase in the film quality by reducing the methane flow during the growth. Single layer with higher homogeneity graphene film was obtained only using 20 sccm CH_4 flow rate.

The reduction in the flow rate of methane leads to minimizing the defects and thus enhancing quality of the graphene film. Due to the high solubility of the carbon in nickel, the higher flow rate of CH_4 caused more carbon accumulation on the substrate, which could be the main reason for the thicker graphene film. Therefore, we can conclude that increasing the carbon source amount during the growth process is an ineffective way to obtain higher quality thin film graphene.

Table 1. Raman spectroscopy results of graphene films grown with different CH_4 flow

SAMPLE NO	FLOW RATE CH_4	I_{2D}/I_G	FWHM
Fig.2a	20 sccm	2,56	30,62
Fig.2b	40 sccm	1,55	29,27
Fig.2c	60 sccm	0,64	42,47
Fig.2d	80 sccm	0,42	75,40

Following the methane flow investigation, the effect of growth time on the graphene synthesis was also examined because the growth time is also one of the most important parameters to synthesize high quality graphene with controlled thickness. Therefore, we synthesized graphene films by changing the growth time from 20 mins. to 50 mins. by 10 mins. increase in each step by keeping all the other growth parameters constant. The Raman spectra of these films grown under the 20 sccm methane are given in Figure 3 and details of the graphs regarding I_{2D}/I_G and FWHM values are provided in Table 2. The I_{2D}/I_G ratio was calculated to be 0,38, 0,71, 1,24 and 2,28 for films grown in 50, 40, 30 and 20 mins., respectively.

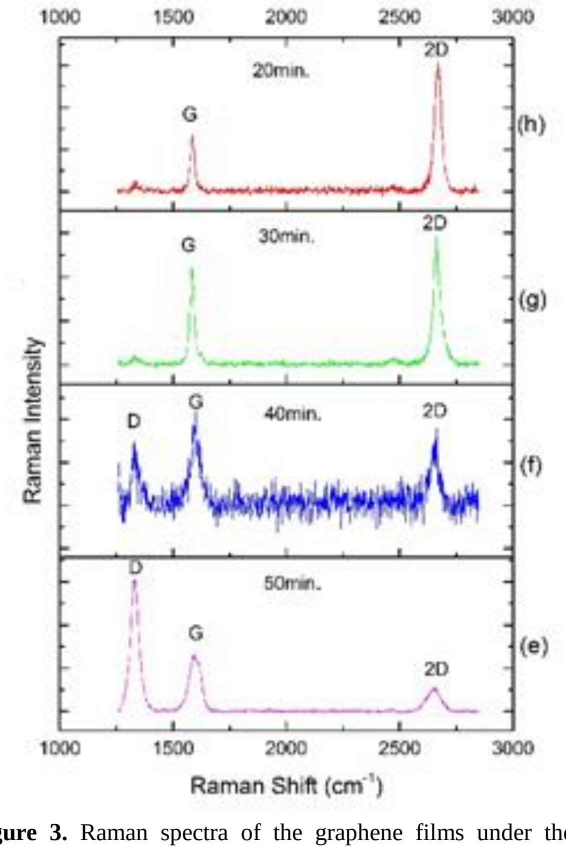

Figure 3. Raman spectra of the graphene films under the various growth time, (e-h) growth time 50-40-30-20 mins. respectively.

The FWHM value was measured as 75,42, 45,71, 33,34, 31,42 for films grown in 50, 40, 30 and 20 mins., respectively. According to these results, multilayer graphene film was obtained with 50 min. growth time and monolayer graphene was grown with 20 min. growth time. Since the carbon solubility in nickel is quite high, to decrease the amount of carbon accumulation in the nickel substrate, the growth time was gradually lowered. Hence, the number of graphene layers were also decreased.

Table 2. Raman spectroscopy results of graphene films grown with different growth time

SAMPLE NO	GROWTH TIME	I_{2D}/I_G	FWHM
Fig.3e	50 min.	0,38	75,42
Fig.3f	40 min.	0,71	45,71
Fig.3g	30 min.	1,24	33,34
Fig.3h	20 min.	2,28	31,42

Graphene film thickness and defects were taken under control on the nickel foil surface by reducing the growth time from 50 mins. to 20 mins. similar to growth time effect. This was expected as the carbon amount in the CVD chamber decreased when decreasing the growth time and methane flow that leads to decrease in the amount of carbon deposited in the nickel foil. Hence, the number of graphene layers was decreased. In our study, the methane flow rate and the growth time showed dramatic effects on the graphene synthesis in terms of thickness and defects. These results are in line with the studies conducted in the literature. Additionally, methane flow and growth time relations on the graphene growth were revealed in this study [14,17].

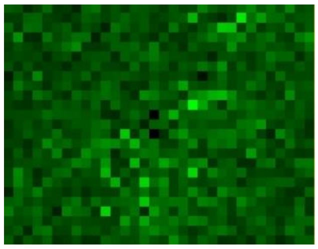

Figure 4. Raman mapping of synthesized graphene with 20 sccm CH_4 and 20 min growth time (100 umx100 um)

The synthesized film was found to be highly homogeneous in terms of thickness as evidenced by Raman mapping (100 umx100 um), which is presented in Figure 4. The map was obtained for the ratio of 2D and G peak intensities at 2700 cm^{-1} and 1585 cm^{-1} positions respectively. The I_{2D}/I_G mapping provided information about the coverage, homogeneity and thickness of the film. The map given in figure 4 confirms the homogeneity and the coverage of the film on the nickel surface. However, although very small, a few black spots in the map indicate the existence of the thicker region in the film and this could be also due to the contamination.

4. CONCLUSION

In this study, we demonstrated that graphene film thickness could be controlled by optimizing methane flow rate and growth time on poly-crystalline nickel substrate in 2-zone CVD system. The synthesized graphene films were characterized using Raman spectroscopy. The number of graphene layer and the film quality were evaluated considering the 2D and G peak positions, their intensity ratios and FWHM of the 2D peak. Based on these results, the flow rate and growth time of methane were verified as important parameters for the homogeneous graphene films as they provided control on the precipitated carbon amount in nickel substrate. Additionally, we found that graphene obtained with 20 sccm methane in 20 minutes growth time was in single layer form with high homogeneity. In addition to the Raman spectra of the films, the Raman mapping, which was obtained for I_{2D}/I_G, was also employed to further confirm the homogeneity of the single layer graphene film.

DECLARATION OF ETHICAL STANDARDS

The authors of this article declare that the materials and methods used in this study do not require ethical committee permission and/or legal-special permission.

AUTHORS' CONTRIBUTIONS

Meryem BOZKAYA: Characterization and writing.

Ali ALTUNTEPE: Methodology, characterization and writing- original draft.

Hakan ATEŞ: Characterization and editing.

Recep ZAN: Conceptualization, methodology and review & editing.

CONFLICT OF INTEREST

There is no conflict of interest in this study.

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