

Investigation of the Effect of Waste Polystyrene Modified with AlCl_3 and FeCl_3 on BPA Removal and Optimisation with CCD-RSM Model

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(Received May 10, 2025; Revised June 4, 2025; Accepted June 5, 2025)

Abstract: In this study, waste polystyrene (PS) was chemically modified using aluminium chloride (AlCl_3) and ferric chloride (FeCl_3) to enhance its adsorption performance for the removal of Bisphenol A (BPA) from aqueous solutions. The modification significantly improved the surface characteristics and adsorption capacity of the polystyrene. The removal efficiency of BPA was systematically evaluated under varying experimental conditions, including initial BPA concentration, solution pH, adsorbent dosage, temperature, and contact time. In order to optimise these parameters, central composite design under response surface methodology (CCD-RSM) was applied to design the experiments: The optimised conditions for the AlCl_3 -modified polystyrene (PS/ AlCl_3) were determined to be; initial concentration 40 mg/L, pH 4, adsorbent amount 0.1 g/L, temperature 40°C and contact time 30 min, while for FeCl_3 -modified polystyrene (PS/ FeCl_3) were initial concentration 40 mg/L, pH 6.3, adsorbent amount 0.1 g/L, temperature 20°C and contact time 90 min. As a result of the adsorption experiments carried out using the developed materials, it was determined that BPA was effectively removed from the simulated wastewater. The maximum adsorption capacities were determined to be 52.17 mg/g for PS/ AlCl_3 and 54.95 mg/g for PS/ FeCl_3 . The results demonstrate that metal chloride modification is an effective approach for valorising waste PS in environmental remediation applications.

Keywords: Waste polystyrene; bisphenol A; CCD-RSM; adsorption; optimization © 2025 ACG Publications. All rights reserved.

1. Introduction

Bisphenol A (BPA) and its phenolic derivatives are considered as an important source of environmental contamination in aquatic environments due to their adverse health effects even at low concentration levels [1]. These compounds are widely used in various industrial activities, such as

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The article was published by ACG Publications

<https://www.acgpubs.org/journal/records-of-agricultural-and-food-chemistry> January-June 2025 EISSN:2792-0763

DOI: <http://doi.org/10.25135/rfac.30.2505.3548>

Available online: June 30, 2025

petroleum refineries, paint production, pharmaceutical manufacturing, and pesticide production, and enter the environment through wastewater from these sectors [2].

There is the possibility of contact with products containing BPA under different environmental conditions [3]. Two main routes of exposure of humans and other organisms to BPA have been identified: direct exposure and indirect exposure. The literature indicates that BPA exposure may contribute to disruptions in the functioning of the endocrine system, increased risk of testicular and brain cancer, neurological and behavioural abnormalities, developmental abnormalities, and triggering the proliferation of cancer cells in humans [4].

Among various removal methods, adsorption is widely recognised as a simple, effective, and sustainable approach for removing pollutants in aquatic environments [5,6]. The adsorption technique, utilising adsorbent materials selected based on parameters such as appropriate pore size distribution, pore volume, specific surface area, and surface properties, offers high potential for this purpose. In this context, it is considered that structural modifications of adsorbents can significantly increase their adsorption capacity by optimising target pollutant-specific interactions [7].

Polymers are widely used as adsorbent materials in wastewater treatment due to their tunable surface chemistry, porous structure, and favourable mechanical properties. Among these polymeric structures, polystyrene (PS) stands out as a promising candidate due to its favourable stiffness, thermal stability, low density, and economic production cost [8]. Therefore, PS has the potential to be widely used as an adsorbent in wastewater treatment processes as well as in various fields [9]. In addition, transforming PS into a high-efficiency adsorbent through chemical modification presents a promising strategy for developing novel materials for wastewater treatment applications [10]. Several pioneering studies have been conducted in this field, laying the groundwork for further advancements in PS-based adsorbent development and wastewater remediation [10-12].

In the present study, waste PS was chemically modified using aluminium chloride (AlCl_3) and ferric chloride (FeCl_3) to enhance its adsorption capacity for removing Bisphenol A (BPA) from aqueous solutions. The structural and morphological characteristics of the unmodified and modified adsorbents were thoroughly analysed using various physicochemical characterisation techniques. The surface modification significantly improved the adsorptive properties of PS, and the performance of the modified materials was systematically evaluated. Key operational parameters—including initial BPA concentration, solution pH, adsorbent dosage, temperature, and contact time—were investigated to determine their influence on removal efficiency. To optimize these parameters and assess their interactive effects, a central composite design (CCD) was employed within the framework of response surface methodology (RSM).

2. Materials and Methods

2.1. Chemicals

The reagents used in the experiments were commercially purchased. Analytical grade hydrochloric acid (HCl) was purchased from Sigma-Aldrich (Steinheim, Germany), PS, AlCl_3 , FeCl_3 , acetone, dimethyl acetal, methanol (CH_3OH), BPA, and dichloromethane (CH_2Cl_2) were purchased from Merck. All chemicals and solvents used were of analytical grade. Pure analytical-grade chemicals were used as received, without any additional treatment. Ultrapure water (18.2 m Ω cm) was used to prepare the working solution in all experiments.

2.2. One-Stage Cross-Linked Polystyrene (PS-FDA) Production

The expanded PS was cut into small pieces (5 g) and placed into a round-bottom flask. First, 400 mL of CH_2Cl_2 and then 56 mL of dimethyl acetal were added to the flask. The mixture was stirred at 300 rpm for 5 min, and AlCl_3 (18.3 g) was added. The mixture was kept at 80°C and stirred (300 rpm) for 12 h. After cooling the mixture, it was evaporated and dried at 50°C for 12 h. The dried product was washed first with a 50% acetone solution (25 mL \times 3) and then with a 1% HCl solution (25 mL \times 3). Acetone, deionised water, and HCl were withdrawn in an evaporator and dried at 50°C for 12 h. The dried material was further purified with CH_3OH at 70°C for 24 h. The CH_3OH was withdrawn in an

evaporator. The obtained precipitate was dried at 50°C for 12 h. The production of progressively cross-linked PS was repeated by adding 22.25 g of FeCl₃ instead of the 18.32 g of AlCl₃ added in the first stage.

2.3. Characterisation

The morphological properties of the adsorbents were investigated using Scanning Electron Microscopy (SEM), (Jeol Jem 7600F model). Fourier Transform Infrared (FTIR) spectroscopy was applied to determine the functional groups using an FTIR spectrometer (Thermo Scientific, Nicolet Is10) in the wavenumber range of 400-4000 cm⁻¹. Ultraviolet-Visible (UV) was performed using UV-Vis spectrophotometer (Perkin Elmer Lambda 35) in the wavelength range of 190-800 nm. pH meter was measured using WTW pH330i model digital.

2.4. Adsorption Experiments and Experimental Design

BPA removal studies were conducted to assess the adsorption efficiency of the adsorbents using the batch equilibration method. The adsorption experiments structured by Central Composite Design-Response Surface Methodology (CCD-RSM) provide an effective approach for modelling and optimization of various parameters affecting the BPA adsorption process [13]. BPA removal rates were determined using a UV-Vis spectrophotometer in the wavelength range of 190-800 nm. The maximum absorbance was observed at 277 nm. The CCD-RSM method applied experimental design principles to improve the removal performance as a response function and to optimise the main parameters affecting this performance. Concentration (A), adsorbent amount (B), pH (C), temperature (D), and contact time (E) are the main parameters of this experimental design. The amounts of 0.1 M NaOH and 0.1 M HCl were used to adjust the pH of the suspension. All adsorption experiments were carried out in an ultrasonic bath. The adsorption capacity (amount of metal ions adsorbed per unit mass) was calculated using Equation 1.

$$q_e = \frac{C_0 - C_e}{m} \times V \quad (1)$$

Here, q_e is the adsorption capacity of the synthesised adsorbent (mg/g), C_0 and C_e are the initial concentration and equilibrium concentration of ions (mg/L), respectively, V is the solution volume (L), and m is the amount of adsorbent (g).

The adsorption experimental design and levels of the independent parameters are presented in Table 1. As shown in Table 1, the CCD method was employed to investigate five coded levels, namely $-\alpha$, -1 , 0 , $+1$, and $+\alpha$, which correspond to the parameter levels. $+\alpha$ of each parameter represents the highest level, 0 represents the middle level, and $-\alpha$ represents the lowest level.

Table 1. CCD coded experimental levels

Parameter	Code	Actual values				
		$-\alpha$	-1	0	$+1$	$+\alpha$
Concentration (mg/L)	A	10	20	30	40	50
Adsorbent amount (mg)	B	0.05	0.1	0.2	0.3	0.4
pH	C	3	4	6	8	10
Temperature °C	D	15	20	30	40	50
Contact Time (min)	E	15	30	60	90	120

3. Results and Discussion

3.1. SEM Images of Adsorbents

The microstructure and morphology of PS/AlCl₃ and PS/FeCl₃ were investigated by SEM. The SEM images revealed significant differences in the morphological properties of both materials (Figure 1). PS/AlCl₃ particles generally consist of irregularly shaped and densely agglomerated structures. The particles have an amorphous or semi-crystalline nature and exhibit a pronounced roughness on the surface [14]. This structure indicates the low crystallisation tendency and rapid particle formation process of PS/AlCl₃. PS/FeCl₃ nanoparticles formed distinctly rod-like or needle-like crystal structures. This structure also facilitates the approach of pollutant molecules to the adsorbent surface [7].

The geometrical shapes of the crystals were more regular and sharp-edged, indicating that PS/FeCl₃ has a high tendency for crystallization. PS/FeCl₃ particles exhibit a heterogeneous structure containing both long crystal rods and smaller particles. As a result, PS/AlCl₃ nanoparticles exhibited a more amorphous and agglomerated morphology, while FeCl₃ nanoparticles formed more distinct crystalline structures, revealing a highly ordered morphology. These differences reflect the different crystal growth mechanisms of both materials, depending on the synthesis conditions and internal structural properties.

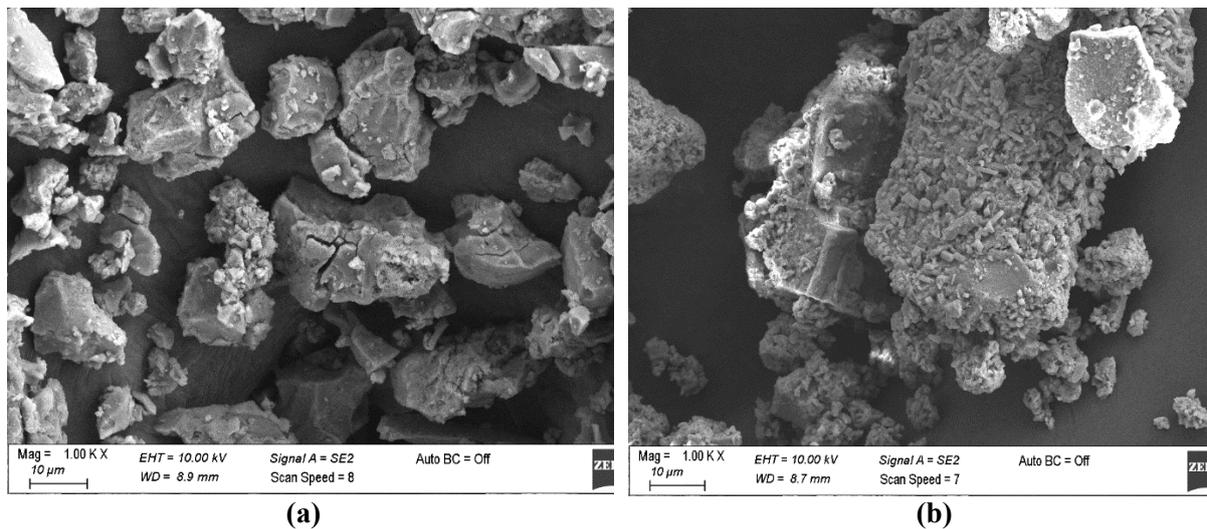


Figure 1. SEM images of (a) PS/AlCl₃ and (b) PS/FeCl₃ adsorbents.

3.2. FTIR Spectra of Adsorbents

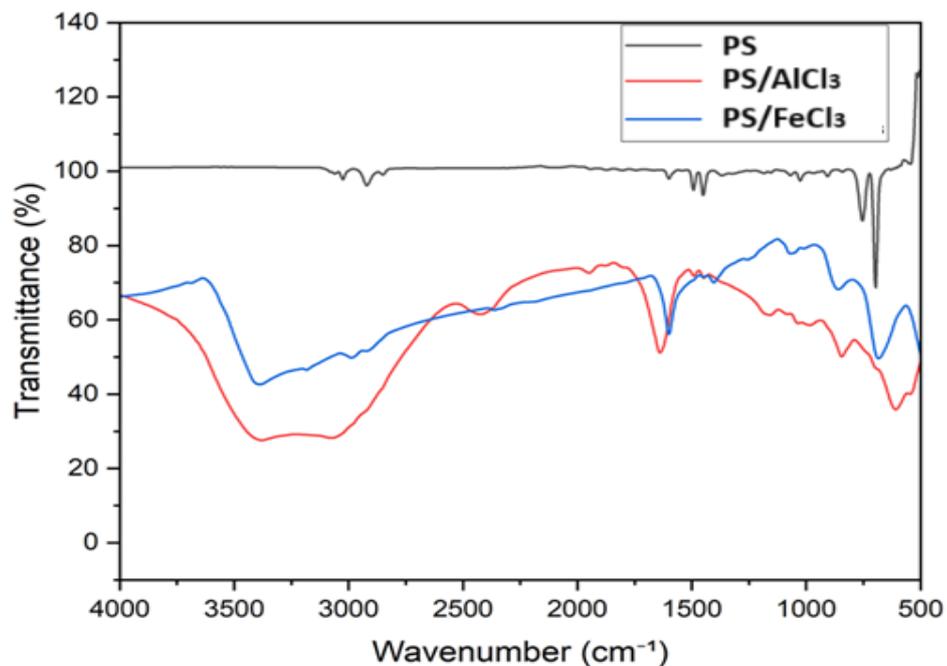


Figure 2. FTIR spectra of the PS, PS/AlCl₃ and PS/FeCl₃ adsorbents.

Table 2. Trial studies designed in CCD for the adsorption capacities for PS/AlCl₃ and PS/FeCl₃

No	Independent Parameters					Adsorption capacity (qe)	
	Concentration (mg/L)	Adsorbent amount (g/L)	pH	Temperature (°C)	Contact time (min)	PS/AlCl ₃ (mg/g)	PS/FeCl ₃ (mg/g)
1	20	0.3	4	20	90	12.29	12.39
2	40	0.1	4	20	30	39.2	38.7
3	30	0.2	3	30	60	31.82	33.35
4	40	0.1	8	40	30	41.97	45.13
5	30	0.05	6	30	60	11.95	52.85
6	40	0.3	4	20	30	28.86	30.94
7	40	0.3	4	20	90	29.1	30.29
8	40	0.1	8	20	90	44.85	52.63
9	20	0.1	4	20	30	42.82	42.88
10	30	0.2	6	30	60	35.78	36.83
11	30	0.2	6	15	60	33.77	35.33
12	20	0.1	8	40	90	39.02	42.18
13	30	0.2	6	30	15	33.26	34.49
14	30	0.2	6	30	60	35.76	36.83
15	20	0.3	8	40	30	11.97	12.38
16	20	0.1	8	40	30	41.15	41.48
17	30	0.2	6	30	120	34.81	35.39
18	40	0.3	8	40	30	39.73	30.57
19	40	0.3	8	40	90	29.45	30.29
20	40	0.1	4	40	90	47.22	52.13
21	40	0.3	4	40	30	29.27	30.37
22	40	0.3	4	40	90	28.64	29.64
23	30	0.4	6	30	60	13.66	14.18
24	20	0.3	8	20	30	12.35	12.47
25	20	0.3	8	20	90	11.61	11.98
26	40	0.1	8	20	30	42.55	50.83
27	20	0.3	4	40	30	11.9	13.12
28	40	0.1	4	20	90	43.4	53.2
29	30	0.2	6	50	60	31.82	33.84
30	20	0.1	8	20	90	40.92	42.35
31	40	0.1	4	40	30	52.17	53.75
32	20	0.1	4	40	30	40.37	41.15
33	50	0.2	6	30	60	49.16	51.71
34	20	0.3	8	40	90	11.61	12.34
35	20	0.3	4	20	30	11.99	11.79
36	40	0.3	8	20	30	29.03	29.6
37	40	0.1	8	40	90	48.55	54.95
38	20	0.1	8	20	30	37.88	40.93
39	20	0.3	4	40	90	11.68	13.33
40	10	0.2	6	30	60	9.7	10.63
41	30	0.2	10	30	60	29.9	33.13
42	20	0.1	4	20	90	40.13	42.63
43	40	0.3	8	20	90	29.6	31.15
44	20	0.1	4	40	90	37.45	40.55

Surface functional groups of PS, PS/AlCl₃ and PS/FeCl₃ were defined by FTIR analysis and shown in Figure 2. When the FTIR spectra was examined, similar peaks (aromatic C-H stretching band at 3000-3600 cm⁻¹, aromatic C=C stretching band at 1600-1700 cm⁻¹) were observed [15]. However, the differences observed

in the intensities of the peaks especially around 1600 cm^{-1} and $1000\text{--}1200\text{ cm}^{-1}$ indicate that structural modifications occur depending on the type or intensity of interactions between the coated metal ions (Al^{3+} and Fe^{3+}) and the polymer matrix [16,17].

3.3. Optimisation of Adsorption Parameters

Forty-four trial studies designed in CCD were completed, and the adsorption capacities for PS/ AlCl_3 and PS/ FeCl_3 were determined (Table 2).

Considering Figure 3, it was observed that the adsorption capacity values increased with increasing concentration of both adsorbent types and then decreased. The concentration effect was studied in the range of 10–50 ppm (Figure 3b). Optimum q_e values were determined as 40 ppm. The saturation of the material surface can express this decrease at high concentrations. Another important parameter, adsorbent amount, was studied in the range of 0.05–0.4 g/L (Figure 3c). It was observed that the removal was higher at a low adsorbent amount (0.1 g/L). At a high adsorbent amount (0.4 g/L), it was observed that the efficiency decreased due to the clustering and agglomeration of the material [18].

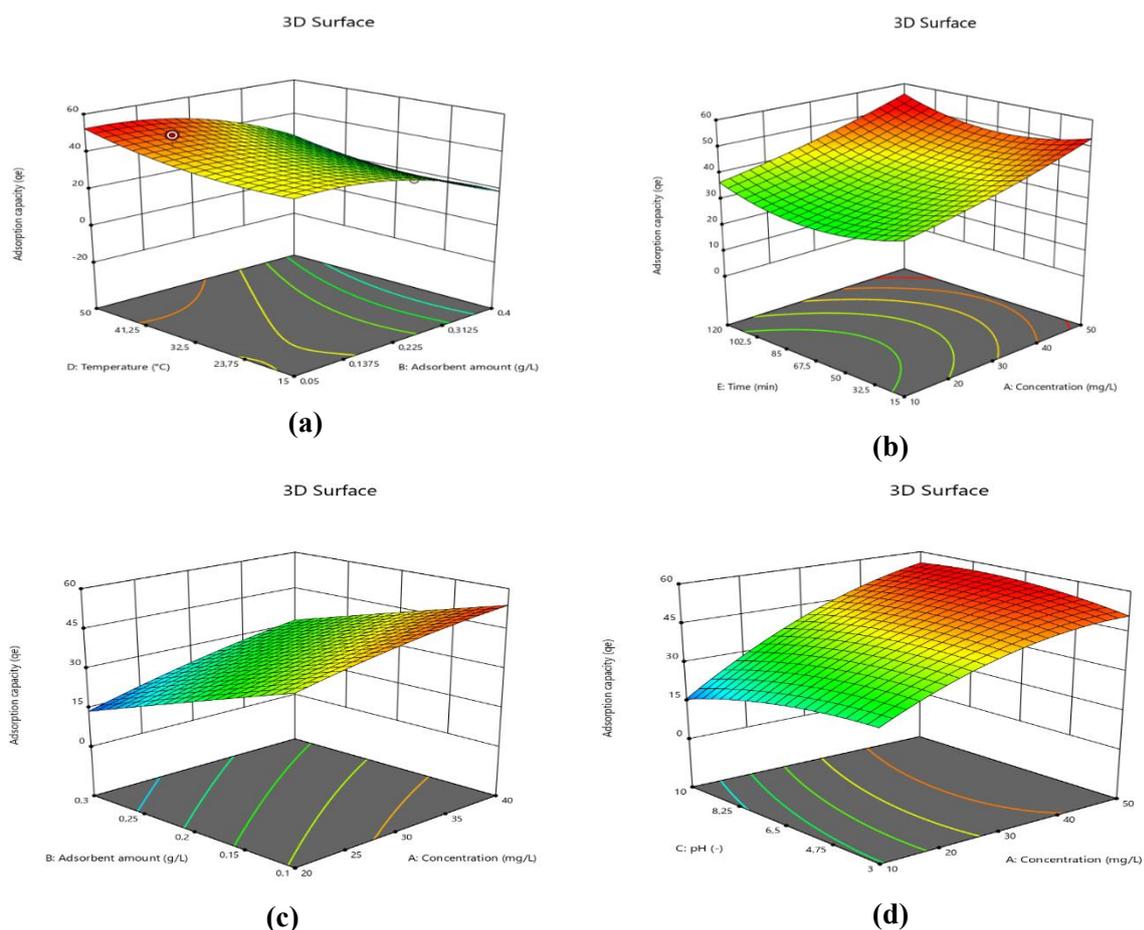


Figure 3. 3D surface plots showing the effects of parameters on the adsorption capacities of PS/ AlCl_3 (a, b) and PS/ FeCl_3 (c, d).

Another parameter, pH effect, was studied in the range of 3 to 10 (Figure 3d). The highest adsorption capacity was achieved at pH 4 for PS/ AlCl_3 , whereas PS/ FeCl_3 was optimized at pH 8. The reason for this is the electrostatic attraction between the positively charged surface and BPA at low pH due to the surface charge of PS/ AlCl_3 [19]. In PS/ FeCl_3 , Fe^{3+} ions can be transformed into hydroxide ions at a pH of 8. As a

result of this transformation, the surface can form stronger complexes with BPA. The temperature effect was studied in the range of 15-50°C (Figure 3a). It was observed that the adsorption capacity increased with increasing temperature. This indicates that adsorption is an endothermic process [20]. The observation of maximum removal at 40°C for both materials indicates that the binding of BPA to the surface requires more energy, and the temperature accelerates this process. Contact time was studied in the range of 15-120 min. While the highest removal in PS/AlCl₃ material occurred at 30 min, the highest removal in PS/FeCl₃ material was observed at 90 min. We can say that at higher contact times, the removal decreases, and simultaneously, the desorption process occurs [21].

In the present study, BPA removal was carried out using adsorbents obtained by modifying waste polystyrene with various metals. The synthesis of materials with large surface area and high active surface sites was targeted and the experimental processes were optimised with the CCD-RSM Design of Experiments model. This approach allowed the systematic evaluation of parameters to improve adsorption efficiency. Although different adsorbents for BPA removal have been developed in the literature, the presented study offers a remarkable alternative in terms of sustainability in terms of reprocessing waste polystyrene and using it for the removal of environmental pollutants. The efficiency of the optimisation-based adsorption method reveals that it has high potential in the control of environmental pollutants and offers an innovative contribution in terms of both waste recovery and pollutant removal when compared with similar studies in the literature.

4. Conclusion

The presented study successfully demonstrated the potential of chemically modifying waste PS material with AlCl₃ and FeCl₃ for BPA removal from aqueous solutions. CCD and RSM determined the optimum BPA removal conditions for both modified adsorbents. The optimum conditions for PS/AlCl₃ material were initial concentration 40 mg/L, pH 4, adsorbent amount 0.1 g/L, temperature 40°C, and contact time 30 min, while for PS/FeCl₃ material, initial concentration 40 mg/L, pH 6.3, adsorbent amount 0.1 g/L, temperature 20°C, and contact time 90 min. Maximum adsorption capacities of 52.17 mg/g for PS/AlCl₃ and 54.95 mg/g for PS/FeCl₃ were reached under optimum conditions. The higher adsorption capacity of PS modified with FeCl₃ suggests that this modification makes the adsorbent surface properties more favourable for BPA interaction. The results obtained demonstrate that waste PS can be transformed into a valuable adsorbent material through appropriate chemical modifications, offering a sustainable solution for preventing water pollution. This study highlights the significance of polymer-based materials in addressing environmental challenges.

Acknowledgments

This study is a part of S. Ö-K's PhD thesis. The authors would like to thank Muğla Sıtkı Kocman University for providing research facilities. We would also like to thank Dilaycan Çam for assisting us with the layout of the journal.

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