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Characterization of Cosmetics Industry Wastewater and Treatment by Coagulation-Flocculation Process

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Kozmetik Endüstrisi Atıksularının Karakterizasyonu ve Koagülasyon-Flokülasyon Prosesi ile Arıtımı

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Öz

Kozmetik ürünlerin üretiminde çok çeşitli hammaddelerin kullanılması ve üretim aşamasındaki sürecin değişkenliği atık su karakterizasyonunu önemli ölçüde etkilemektedir. Kozmetik endüstrisi atıksuları biyolojik olarak parçalanabilirliği düşük bileşenler içermekle birlikte; amonyak, yağ ve gres, fosfor ve ağır metaller gibi çeşitli kirleticileri de içerebilmektedir. Kozmetik endüstrisinde bulunabilen ağır metaller kansere, nörolojik hastalıklara, genetik mutasyonlara, saç dökülmesine ve doğum kusurlarına neden olmaktadır. Bu çalışmada, gerçek kozmetik endüstrisi atıksuyundan alınan numunelerde pH, Kimyasal Oksijen İhtiyacı (KOİ), Toplam Azot (TN), Toplam Fosfor (TP), Askıda Katı Madde (SS), Bakır (Cu), Çinko (Zn), renk ve Ortofosfat (PO_4-P) analizleri yapılarak, kanalizasyon deşarj kriterlerine uygunluğu araştırılmıştır. Kirleticilerin giderimi koagülasyon-flokülasyon prosesi ile araştırılmış ve giderim verimleri KOİ: %75; Cu: %92; Zn: %94; Renk: %84; TN: %99; TP: %91; PO_4-P: %92 olarak bulunmuştur. Yüksek KOİ giderimi elde etmek için farklı arıtma proseslerinin araştırılması gerekmektedir. Çevre ve insan sağlığının korunması için gerçek kozmetik endüstrisi atıksularının karakterize edilerek ağır metallerin belirlenmesi ve giderimine yönelik çalışmaların arttırılması önemli olacaktır.

Anahtar Kelimeler: *Kimyasal arıtma; koagülasyon-flokülasyon prosesi; kozmetik atıksuları; organik kirleticiler; ağır metaller.*

Abstract

The use of a wide variety of raw materials in the production of cosmetic products and the variability of the process during the production phase significantly affect wastewater characterization. Cosmetic industry wastewater contains low biodegradable components and may contain various pollutants such as ammonia, oil and grease, phosphorus and heavy metals. Heavy metals that can be found in the cosmetics industry cause cancer, neurological diseases, genetic mutations, hair loss and birth defects. In this study, pH, Chemical Oxygen Demand (COD), Total Nitrogen (TN), Total Phosphorus (TP), Suspended Solids (SS), Copper (Cu), Zinc (Zn), color and Orthophosphate (PO_4-P) analyses were carried out in samples taken from real cosmetic industry wastewater and their compliance with sewage discharge criteria was investigated. Pollutant removal was investigated by coagulation-flocculation process and removal efficiencies were found as COD: 75%; Cu: 92%; Zn: 94%; Color: 84%; TN: 99%; TP: 91%; PO_4-P: 92%. In order to achieve high COD removal, different treatment processes should be investigated. In order to protect the environment and human health, it will be important to increase the studies on the determination and removal of heavy metals by characterising the real cosmetics industry wastewater.

Keywords: *Chemical treatment; coagulation- flocculation process; cosmetic wastewaters; organic pollutants; heavy metals.*

1. Introduction

The growth in population and industrialization, various facilities have been established in different sectors to meet human needs. With increasing and diversifying industrialization, pollutant parameters are differentiated and wastewater with different characteristics are generated. Since access to potable and usable water is becoming more and more difficult, it is inevitable to treat the wastewater (Karakas *et al.* 2020; Kizil and Sonmez, 2022; Obotey Ezugbe and Rathilal, 2020). Cosmetic ındustry have one of devleoping sectors, which lead to the production of different types of products in industry. At the same time, environmental concern have been raised due to the increasing production of cosmetic wastewater.(Can *et al.* 2024).

In the cosmetics industry; various products such as shampoo, conditioner, hair dye, sunscreens, moisturizers are produced and wastewater is generated from the production of cosmetic products (Bayhan and Değermenci, 2017). Raw materials used in the production of cosmetic products include water, detergents, emulsifiers, dyes, organic solvents and essential oils (Lima *et al.* 2022; Turkay and Kizil, 2024). The steps followed in the production of cosmetic products include receiving,

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storing, weighing and sorting of raw materials, production, physicochemical and microbiological analysis, filling and packaging (Bom *et al.* 2019).

Cosmetic industry wastewater is generated as a result of cleaning and sterilizing equipment, instruments and floors and contains mostly organic matter, oil and grease, sulfides, ammonia, surfactants, phosphates and polyphosphates (Abidemi *et al.* 2018). Since cosmetic industry wastewater contains high concentrations of phosphates and polyphosphates, eutrophication can occur in receiving environments where the wastewater is discharged (Muisa *et al.* 2020). Substances such as paraben and triclosan, which are used as preservatives in cosmetic products, could be found in cosmetic industry wastewater and pose an environmental risk. These components can cause toxic effects on aquatic organisms and have negative effects on the environment and human health (Bilal *et al.* 2020). In addition, heavy metals are used as colorants or preservatives in cosmetics products and can damage the kidneys and nervous system (Mohammed *et al.* 2023; Raza-Naqvi *et al.* 2022). For instance, high concentrations of Cu in cosmetic wastewater are toxic and carcinogenic, causing respiratory problems, liver and kidney failure and gastrointestinal bleeding. Moreover, high concentrations of Zn can cause various health problems such as fever, stomach cramps, vomiting, anaemia and nausea, and can damage the kidney, liver and immune system (Abed *et al.* 2023).

In order to remove contaminants in wastewater, different processes have been applied for treatment of cosmetic wastewater (Akbay *et al.* 2022). These processes include coagulation (Michel *et al.* 2019), advanced oxidation (Naumczyk *et al.* 2014), membrane filtration (Huisman, 2004), membrane bioreactor (Monsalvo *et al.* 2014; Zhang *et al.* 2022), anaerobic bioreactor (Wiliński *et al.* 2017). Coagulation-flocculation is recognized as one of the most fundamental methods for wastewater treatment (Ahmad *et al.* 2024; Simate, 2015). The coagulation-flocculation process is to improve water quality by removing colloidal or suspended particles (Abu-Dalo *et al.* 2022). It is stated as a highly efficient and environmentally friendly process in terms of pollutant removal. It is an attractive option for wastewater treatment due to its easy operation and low cost (Fard *et al.* 2016; Kurniawan *et al.* 2023; Zhao *et al.* 2021). In the literature, $Al_2(SO_4)_3$, polymerized aluminum chloride, $FeSO_4$ and $FeCl_3$ were used as coagulants in the treatment of cosmetic industry wastewater and Chemical Oxygen Demand (COD) removal efficiency was found 75% (Naumczyk *et al.* 2014). It was reported that the removal of anionic surfactant was not effective (24%) in the coagulation process with $Al_2(SO_4)_3$ and $FeCl_3$ (Kaleta and Elektorowicz, 2013). In the coagulation process using $FeCl_3$, tonalide, galaxolide and celestolide removal in cosmetics industry wastewater was 83.4%, 79.2% and 77.7%, respectively (Suarez *et al.* 2009).

Detailed characterization studies using real cosmetic industry wastewater are insufficient in the literature. In addition, there are also insufficient studies in which the removal efficiencies of pollutant parameters are comprehensively investigated in Türkiye. The aim of this study was to supply characterization of cosmetic wastewaters with different pollutant composition and concentration, to extend the knowledge about these wastewaters and to investigate the removal of pollutants by coagulation-flocculation process.

In this study, the real wastewater was taken from the cosmetics industry in the Gebze Organised Industrial Zone (GOIZ) and the treatability of wastewater by coagulation-flocculation process was investigated using iron III chloride ($FeCl_3$), lime ($Ca(OH)_2$) and anionic polyelectrolyte ($(NH_4)_2S_2O_8$). Chemical Oxygen Demand COD, pH, Total Nitrogen (TN), Total Phosphorus (TP), Suspended Solids (SS), Copper (Cu), Zinc (Zn), color, and Orthophosphate (PO_4-P) parameters were monitored in wastewater. The suitability of effluent concentrations after coagulation-flocculation process in terms of sewage discharge criteria was investigated and removal efficiencies of pollutant compounds were determined.

2. Materials and Methods

2.1. Description of the Cosmetics Wastewater Treatment Plant and Sample Collection

The cosmetics industry is located GOIZ in Türkiye. The facility produces cosmetic products such as face, eye and nail care products and make-up materials. The facility is located in a total area of 7840 m^2. A total of 4m^3/day of wastewater is generated as a result of production. Homogeneous mixing of industrial wastewater is ensured in the equalization basin by aeration. The wastewater is treated with cationic polyelectrolyte and then oil are removed in the DAF unit. Acid cracking, coagulation and flocculation processes are applied to the wastewater respectively. In the first stage of the coagulation process, $Ca(OH)_2$ is added to the wastewater and in the second stage, $FeCl_3$ is added and rapid mixing is performed. After the coagulation process, $(NH_4)_2S_2O_8$ is added to the wastewater and the flocculation process is completed by observing a dense floc formation in the tank with slow mixing. Flocs are kept in the chemical settling tank for 2-4 hours. After chemical precipitation, the treated upper phase is taken from the wastewater and discharged to the sewage. Figure 1 shows the stages used in the cosmetics industry.

2.2. Sample Collection

Samples were taken three times a week and a total of 2 liters of composite samples were created, half a liter sample per hour. The obtained wastewater from the different stages used in production and stored at 4 °C in refrigerator before the analyses.

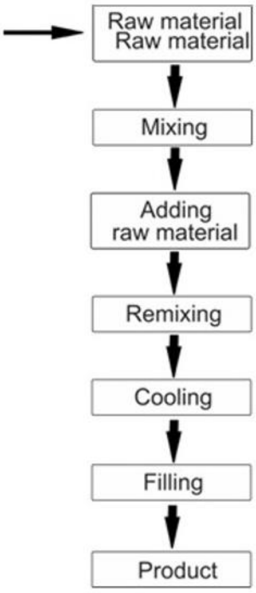

Fig. 1. Formation stages of wastewater in the cosmetics industry

2.3. Analysis Method

The COD, SS, Heavy metals (Cu and Zn), Color, TN, TP, PO_4-P and pH parameters are monitored in the raw and treated wastewater. The pH was measured using a pH meter. COD (K-7375), Cu (LCK-329), Zn (LCK-360), TP (TR-TP), TN (TR-TN) and PO_4-P (TR-PO_4) were carried out with Hach test kits. SS were determined according to Standard Methods (1994) (American Public Health Association, 1995). Color was analyzed using Hach lange DR300 spectrophotometer.

2.4. Coagulation-Flocculation Jar Test Procedure

Coagulation-flocculation study was carried out under laboratory conditions (24 °C) using a jar test device. During the research, 200 mL wastewater volume was used in the study. Each coagulation experiment was started by adding coagulant to the reactor containing wastewater, and then the appropriate initial pH was adjusted using HCl and NaOH. In the coagulation step, the reaction time was limited to 3 minutes and the process was continued at a stirring speed of 120 rpm (El Gaayda *et al.* 2023). The flocculation process was carried out at a mixing speed of 30 rpm with a contact time of 10 minutes (Kilic and Kumbasar, 2023). The precipitation step was determined as 45 minutes and at the end of the process, samples were taken from the upper phase and analyzed for COD, SS, Cu and Zn, Color, TN, TP, PO_4-P. The properties of the chemicals used are presented in Table 1 and Table 2. The values given in Table 3 were found using Table 1 and Table 2. The concentrations of coagulant used in the influent wastewater and the optimum pH values were determined at three different sets by Jar test in Table 3. Images of all jar tests applied to cosmetic industry wastewater is given in Figure 2. The percentage removal efficiency (E) was calculated according to the equation:

$$E(\%) = (C_0 - C)/ C_0 * 100 \quad (1)$$

where: C_0 and C are, respectively, the initial and final values, as determined in raw wastewater (influent) and decanted supernatant liquid after coagulation-flocculation (effluent).

3. Results and Discussions

The parameters and concentrations monitored in cosmetics industry wastewater vary according to the process and the studies are summarized in Table 4. Cosmetics industry effluent is aimed to ensure GOIZ sewage discharge criteria. GOIZ sewage discharge limit values of treated wastewater and the characterization of real cosmetic industry wastewater is summarized in Table 5.

The standard for discharge to sewage between all jar tests was achieved with chemical concentrations applied in set 3. In the cosmetic industry wastewater 5% $Ca(OH)_2$, 40% $FeCl_3$ and 0.1% $(NH_4)_2S_2O_8$ in coagulation-flocculation. In the coagulation flocculation process, 1.250 mL $FeCl_3$, 0.170 g $Ca(OH)_2$ and 0.011 g $(NH_4)_2S_2O_8$ were applied in Set 3, respectively in Table 3.

Table 1. Properties of the chemicals used

	Coagulants and Chemicals					
	HCl	**NaOH**	**$FeCl_3$**	**$(NH_4)_2S_2O_8$**	**H_2SO_4**	**$Ca(OH)_2$**
Concentration	37%	45%	40%	0.1%	98%	5%
Density (g/cm^3)	1.180	1.32	1.435	0.75	1.83	2.21
Mole (g/mole)	36.5	40	162	228	98.08	74.09
Molar concentration (mol/m^3)	11.962	14.850	3.543	0.003	18.285	1.491

Table 2. Consumption of coagulants according to set 3 for 200 mL sample

Coagulants	Concentration	Consumption (mL)	Density(d) (g/cm^3)	Consumption (g)
$FeCl_3$	%40	1.250	1.435	0.718
$(NH_4)_2S_2O_8$	%0.1	-	0.75	0.011
$Ca(OH)_2$	%5	-	2.21	0.170

Table 3. Coagulants used in the study, effective volume, doses and pH values

		Doses			pH values		
		Set 1	Set 2	**Set 3**	Set 1	Set 2	**Set 3**
	Effective volume in sets	(100 mL)	(100 mL)	(200 mL)	(100 mL)	(100 mL)	(200 mL)
Coagulants and Chemicals	HCl (mL)	1.050 mL	1.050 mL	-	1.97	1.97	-
	NaOH (mL)	0.800 mL	0.800 mL	-	11.02	11.02	-
	$FeCl_3$ (mL)	1.200 mL	1.200 mL	**1.250 mL**	8.47	8.47	**4**
	$(NH_4)_2S_2O_8$ (g)	0.053 g	0.008 g	**0.011 g**	8.47	8.47	**8.61**
	$Ca(OH)_2$ (g)	**-**	**-**	**0.170 g**	**-**	**-**	**8.61**

Table 4. Raw wastewater characteristics from cosmetic industries

	Parameters				Reference
pH	**COD (mg/L)**	**TN (mg/L)**	**TP (mg/L)**	**SS (mg/L)**	
1-12	1000-5000	-	-	450-1440	(Araujo *et al.* 2008)
7.1	9700	-	-	-	(Alves, 2009)
6-8	4000-15000	-	-	80-160	(de Oliveira *et al.* 2009)
5.5	13570	107.37	-	2340	(Friha *et al.* 2014)
7.2	15339	-	1.4	-	(Marchetti, 2014)
4.52	7940	-	-	1900	(Bayhan and Değermenci, 2017)
11.4	7125	-	-	-	(de Melo *et al.* 2018)
7.27	1140	-	-	18	(Muszyński *et al.* 2019)
-	1042	-	-	22	(Reinehr *et al.* 2019)
-	5960	-	0.4	-	(Araújo *et al.* 2022)
11.8	**9160**	**81**	**19.3**	**3000**	**Present Study Value**

pH, COD, TN, TP, SS, Cu, Zn, Color, and PO_4-P parameters were monitored in real cosmetic industry influent wastewater. In this study, the concentration of COD in the influent wastewater was found 9160 mg/L in accordance with the literature (de Oliveira *et al.* 2009). While Friha have found TN as 107.37 mg/L in the study, it was found as 81 mg/L in this study which close to the literature (Araujo *et al.* 2008). Also, TP concentration was found 19.3 mg/L in influent wastewater, while TP concentrations in the literature were found very low at 1.4 mg/L (Marchetti, 2014) and 0.4 mg/L (Araujo *et al.* 2008). SS concentration was determined as 3000 mg/L in present study, which was higher than the values given in the literature. In addition, the pH value is 11.8 in influent wastewater in this study is similar to the literature (Araujo *et al.* 2008; de Melo *et al.* 2018). When heavy metal and PO_4-P concentrations are considered in influent wastewater; Cu, Zn and PO_4-P concentration were determined as 6.07 mg/L, 3.5 mg/L and 6.25 mg/L, respectively. The color was found to be 880 Pt-Co. Cu, Zn, Color, and PO_4-P parameters were not available in the literature.

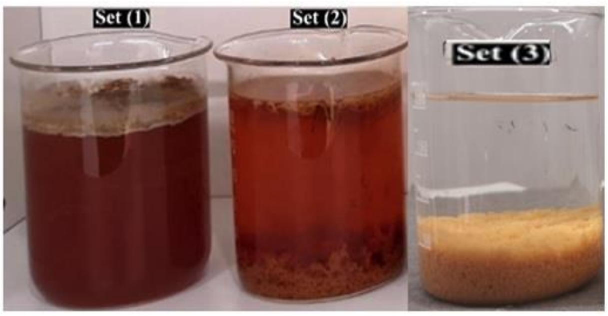

***Fig. 2**. Jar test images*

Table 5. Influent and Effluent concentrations, removal rate and discharge criteria

Parameter	Influent concentrations	Effluent concentrations	Removal Rate (%)	Discharge criteria*
COD (mg/L)	9160	2260	75	4000
pH	11.8	8.8	-	6.5-10
SS (mg/L)	3000	<50	-	500
Cu (mg/L)	6.07	0.48	92	2
Zn (mg/L)	3.5	0.22	94	10
Color (Pt-Co)	880	144	84	-
TN (mg/L)	81	1	99	80
TP (mg/L)	19.3	1.8	91	2
PO_4-P (mg/L)	6.25	0.5	92	-

***** Discharge criteria: GOIZ discharge criteria *(Water Pollution Control Regulation, Table 25)*

In this study, three jar tests were performed. As can be seen from Figure 2, the image with the best collapse in the jar test results was the Set 3. The results obtained from the first two jar tests did not provide optimum settling and removal of color. In addition, the standard for discharge to sewage could not be achieved in these results. The influent wastewater concentration, jar test results in Set 3 (effluent concentration) and removal efficiencies are given in Table 5.

COD, Cu, Zn, Color, TN, TP and PO_4-P concentration and removal rate were monitored in present study. COD removal was found 75% as seen in Table 5. COD removal from dye industry wastewater was investigated using $FeCl_3$ and dragon fruit peel. COD removal was determined between 38% and 46% using 5 mg/L $FeCl_3$ and dragon fruit peel at precipitation time of 40 min and pH 6.0 (Nguyen *et al.* 2022). In the removal of dye from industrial wastewater by coagulation-flocculation, 98% COD removal was determined at pH 11.25-11.7 using 4.07-4.25 mL $AlCl_3$ (Harfaoui *et al.* 2023). In a different study, 67% COD removal was determined in the treatment of olive mill wastewater by coagulation-flocculation using 5 g/L lime and 4 g/L alum (Fleyfel *et al.* 2024). The effect of pH on COD removal was investigated and COD removal was 32% at pH 9.0 in the coagulation process using $FeCl_3$. The lowest removal efficiency was observed 20% at pH 6.0 (Naumczyk *et al.* 2014). Similarly, using physicochemical treatment processes COD removal rate was 36.5% at hair care products wastewaters (de Melo *et al.* 2013) and 31.7% (Comin, 2017) in the literature.

It is reported that coagulation is very effective in COD removal and up to 90% COD removal is achieved in the literature. In a study using cosmetics industry wastewater, COD removal efficiency was found in the range of 13-21% as a result of the coagulation process. The low removal efficiency was associated with the lack of suspensions and colloids in the wastewater (Muszyński *et al.* 2019). It was clearly seen that it was above the COD removal efficiencies in the literature in this study. Since the main purpose of this study was to supply the criteria for discharge to sewage, the effluent wastewater concentration of 2260 mg/L was suitable for the criteria.

Cu was found above the discharge criteria for sewage while Zn was found below the discharge criteria in the influent wastewater. Cu and Zn compounds removal efficiencies were found 92% and 94%, respectively. Zn removal was found to be 63% and copper removal was found to be 77% as a result of coagulation-flocculation treatment of textile industry wastewater using aluminium sulphate (Bouaouine *et al.* 2017). In another study investigating copper and zinc removal from palm oil industry wastewater using moringa as a coagulant, removal efficiencies were found to be 94% and 82%, respectively (Jagaba *et al.* 2021). The results obtained for Cu and Zn compounds in this study are in parallel with the literature. In this study, Cu and Zn compounds in the effluent wastewater concentration supplied the discharge limits.

In this study the color removal was found to be 84%. The color in the effluent was determined as 144 Pt-Co. In the coagulation process using aluminum sulfate, the color removal efficiency was 93.63% (Araujo *et al.* 2008). Removal of indigo blue dye from an industrial wastewater using $AlCl_3$ by coagulation-flocculation process was investigated. Color removal was found to be 98.21% at pH 11.25, using coagulant dose of 4.07 mL and flocculant dose of 0.94 mL (Harfaoui *et al.* 2023). In the coagulation flocculation process using plant-based and chemical coagulants, color removal was 59.42% and COD removal was 54.63% with the addition of 0.79 mg/L plant-based coagulant. In the same study, colour removal was 97.29% and COD removal was 75.31% with the addition of 698.4 mg/L alum (Ahmad *et al.* 2024). In another study, the combination of coagulation-flocculation using PACl and hydrodynamic cavitation with O_3 was evaluated for the treatment of textile wastewater. The color removal was

92.9% and COD removal was 84.1% (Marques *et al.* 2025). In the treatment of textile wastewater, colour removal was 59.6% and COD removal was 70.7% using PGα21Ca in coagulation-flocculation process (Mohanraj *et al.* 2023). It is seen that the color removal in this study is compatible with the literature.

TN removal efficiency in cosmetic industry wastewater was found to be 99% in this study, TN was almost completely removed by supplying the discharge limit. In Set 3 with the coagulation flocculation process carried out with the chemicals applied, sewage discharge limit values were supplied for TP. In this study, TP effluent concentration was found 1.8 mg/L and removal efficiency was 91%. Pretreatment of landfill leachate by coagulation-flocculation was investigated. Under optimum conditions (polymeric iron sulfate: 12.18 g/L, initial pH: 8.12 and polyacrylamide: 11.29 mg/L), COD removal was 62.30 % and TP removal was 95.79 % (Zhang *et al.* 2023). The PO_4-P concentration in the effluent was 0.5 mg/L and the removal efficiency was found 92%. Also, there is no limit value determined for PO_4-P among GOIZ sewage discharge standards. As seen in Table 5, all of the pollutants detected in this study and included in the Water Pollution Control Regulation supplied the discharge criteria.

4. Conclusions

The cosmetic industry wastewater, which varies greatly in terms of pollutant type, was characterized and effluent wastewater found to be in compliance with sewage discharge standards by using coagulation-flocculation process. In addition, the removal of COD, Cu, Zn, Color, TN, TP and PO_4-P was investigated. In this study, while COD removal rate was found 75%; Cu and Zn removal efficiency was 92% and 94% and Color, TN, TP and PO_4-P removal rate are ≥84%.

In this study, different treatment processes should be investigated for more effective COD removal. Further studies and detailed characterization of cosmetic industry wastewater are needed to better characterize the environmental risks posed by cosmetic industry wastewater and to determine appropriate treatment processes. Additionally, when investigating the characterization of cosmetic industry wastewater, examining parameters such as sulfides, oil-grease and conductivity may be important in determining appropriate treatment processes. It is important to detail production processes in the cosmetics industry to reduce water consumption and treat wastewater with high treatment performance. The diversity of raw materials to be used and the differences in production processes lead to the formation of wastewater that can vary widely. The composition and concentration of pollutants in wastewater from the cosmetics industry can vary depending on changes in production and time. The pollutant concentration of these wastewaters is usually high and resistant to biological treatment and the pollutant concentration is usually high. Determining sustainable treatment technologies will be important in terms of protecting water resources and removing treatment-resistant pollutants can be achieved.

Declaration of Ethical Standards

The authors declare that they comply with all ethical standards.

Credit Authorship Contribution Statement

Author-1: Sources, Research, Experiment, Methodology, Writing- original draft Visualisation, Writing- original draft.

Author-2: Research, Formal analysis, Verification, Visualisation, Writing- original draft.

Declaration of Competing Interest

The authors have no conflicts of interest to declare regarding the content of this article.

Data Availability Statement

All data generated or analyzed during this study are included in this published article.

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