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MICELLE-MEDIATED EXTRACTION COMBINED WITH THE ADSORPTION ONTO MAGNETIC NANOPARTICLES FOR THE EXTRACTION OF CRYSTAL VIOLET FROM WASTEWATER

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ABSTRACT. This work describes cloud point extraction combined with the adsorption onto magnetic nanoparticles (MNPs) for separation and spectrophotometric determination of crystal violet (CV). Briefly, CV molecules were adsorbed on MNPs and transferred into the surfactant-rich phase of Triton X-114. Subsequently, the target analyte was desorbed from MNPs by acidified ethanol and detected spectrophotometrically. The impact of experimental conditions, including pH, electrolyte, amount of Triton X-114 and MNPs, incubation temperature and time, type, and volume of the eluent was investigated. The calibration curve was linear in the range of 1.5-250.0 μgL^{-1} of CV. A preconcentration factor of 42.9 was obtained, and the detection limit for CV was 0.5 μgL^{-1}. The intra-day and inter-day relative standard deviations (RSD) for five replicate analyses of CV (100 μgL^{-1}) were 1.7% and 2.1, respectively. The developed approach was effectively used for quantitative analysis of CV in environmental water samples with accepted recovery (97.0-101.2%). The procedure is accurate, precise, rapid and low-cost.

KEY WORDS: Cloud point extraction, Solid phase extraction, Fe3O4 nanoparticles, Crystal violet, Wastewater samples

INTRODUCTION

The effusion of pollutants from wastewater is currently one of the world's most serious issues. Textile industries contribute significantly to the global economy and environmental damage. Textile effluent includes dyes, heavy metals, surfactants, and other chemicals that are often highly poisonous, non-degradable, and difficult to remove using traditional wastewater treatment procedures, and therefore they pose significant threats to ecosystems and humans [1-3].

Crystal violet (CV) is a basic (cationic) dye employed in many dyeing industries and can persist in the environment due to its resistance to biodegradation. Human toxic effects of CV depend on the route of administration and include eyes, skin, and gastrointestinal tract irritation [4]. On the other hand, the presence of pigments in water sources blocks light from marine organisms leading to the scarcity of life in these places [5]. Therefore, it is necessary to handle effluents containing dyes to provide a safe and clean environment [6, 7].

Several techniques have been employed to remove dyes, including biodegradation [8], ultrafiltration [9], reverse osmosis [10], ozonation [11], and adsorption [12]. Due to their simplicity, low cost, and flexibility, adsorption-based treatments (solid phase extraction, SPE) have become increasingly prevalent in recent years [13]. For this purpose many adsorbents , such as active carbon [14], clays [15], metal oxide nanoparticles [16], and nanocomposites [17] have been used. Among the adsorbents, magnetic nanoparticles (MNPs) have become of major interest owing to their excellent adsorption properties as well as the capacity to separate quickly and easily by applying external magnets [18]. To date, iron oxide nanoparticles (such as magnetite, Fe_3O_4) have been the most widely accessible MNPs. Significant advancements in MNPs have been achieved to increase their effectiveness in treating water using various techniques, including

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*Corresponding authors. E-mail: w.mortada@mans.edu.eg chemical functionalization, or doping with other active molecules [19-21]. Despite many benefits and advantages of the SPE, it suffers from some shortcomings, such as time-consuming, expensive, agglutination of sorbent, relatively low reproducibility, poor dispersibility, and disposal of hazardous solvents [22].

Most of the disadvantages of SPE could be solved by employing surfactants, which have the properties of contact at the liquid-solid interface and are arranged into supramolecular structures, resulting in stable colloidal dispersions [23]. Adsorption of surfactant molecules onto the surface of the employed sorbent causes steric or electrostatic repulsions, which improve their wetting properties [24]. Surfactants can also help separate the solid sorbent from the aqueous medium by causing clouding above a certain temperature, which is the foundation of the eco-friendly technique of cloud point extraction (CPE) [25-27]. The combined use of CPE approach and adsorption onto multi-walled carbon nanotubes enables the effective removal of Rhodamine B from water samples [28]. In a recent study, Abdelwahed *et al.* applied a combined CPE and SPE using montmorillonite as natural adsorbent for preconcetration of lead ions prior analysis of food samples [29]. A novel spectrophotometric technique for determining methylene blue in water samples has been developed. The approach is based on a combination of dispersive SPE and CPE with $Cu(OH)_2$ nanoflakes as an adsorbent [30]. The incorporation of magnetic nanoparticles (such as nickel/zinc ferrite) in CPE reduced extraction time by eliminating the centrifugation and freezing steps required for the traditional CPE. The approach was applied for spectrofluorometric determination of citalopram, an anti-dipressive drug, in biological and pharmaceutical samples [31]. A coupled magnetic SPE-CPE employing reduced graphene oxide-Co_3O_4 nanocomposite as an effective adsorbent was employed for the separation, and determination of Sunset Yellow in food samples by spectrofluorometry [32].

The major purpose of this work was to explore the feasibility of employing CPE in the presence of MNPs (Fe_3O_4) to remove CV from aqueous solutions. The approach combines the benefits of adsorption onto MNPs with concentration of the target dye into the surfactant-rich phase, leading to effective and rapid removal. To achieve high extraction recovery of CV, the suggested approach was adjusted for pH, amount of Triton X-114, mass of MNPs, sample volume, and extraction time. To the best of our knowledge, no earlier studies have described the advantages of using the CPE/MNPs system for dye removal from water samples.

EXPERIMENTAL

Chemicals

Analytical-grade chemicals were utilized in the work. MNPs (Fe_3O_4, 5 nm diameter) were purchased from Nanotech Egypt for Photo-electronics Co. (6th of October City, Egypt). A 1000 mg L^{-1} solution of CV (Alpha Chemica, India) was prepared by dissolving 1.0 g in 1000 mL of deionized water. Working solutions of CV were prepared by serial dilution of this stock solution. Triton X-114, octyl phenol polyethylene glycol ether, was obtained from Sigma (St. Louis, MO, USA). Acetate buffer (pH 4.0-5.0) and phosphate buffer (6.0-8.0) were used to examine the influence of pH on the extraction and determination of CV.

Instrumentation

Absorbance was measured using a 7305 Genway UV-Vis spectrophotometer (Staffordshire, UK) at 590 nm. The pH was determined using a digital pH meter (Hanna Instruments Inc, RI, USA). During the extraction procedure, a thermostat bath (Memmert, Germany) was used for temperature control.

Optimization procedure

The factors affecting the performance of the extraction procedure were investigated and optimized to achieve a good recovery, by using the univariate approach which means changing one parameters at a time and keeping all other variables constant [33]. Table 1 illustrates the studies range for each variable.

Table 1 Studies range for experimental variable during optimization of the extraction procedure.

Variable	Studies range
pH	4.0 – 8.0
Concentration of Triton X-114	0.05 – 0.15 v/v %
Mass of Fe_3O_4	2.0 – 6.0 mg
Equilibrium temperature and time	25.0 – 55.0 °C / 10 – 30 min
Eluent type	Ethanol, ethanol/HNO_3 (5:1 v/v), acetone, NaOH (0.1 mol L^{-1})
Eluent volume (ethanol/HNO_3 5:1 v/v)	250 – 500 µL

Extraction procedure

An aliquot (15.0 mL) of 10 mg L^{-1} of CV was mixed in a flask with 4.0 mg of MNPs and Triton X-114 at the required pH. The suspension was then stirred for 1.0 min by a vortex to disperse the MNPs into the solution and incubated in the thermostat water bath at 45 °C for the desired time. At this stage, a cloud solution was obtained. After that, the MNPs were collected at the side of the tube by an external magnet. The separated CV onto MNPs was eluted by ultrasonication with 350.0 µL of ethanol/HNO_3 mixture (5:1) for 5.0 min. Then, the MNPs were separated from the eluent by the magnet and the eluent was centrifuged and analyzed for CV at 590 nm by UV-Vis spectrophotometer. The extraction recovery was calculated using the following equation:

$$R\ (\%) = \frac{\text{Measured concentration of extracted CV}}{\text{Concentration of added CV}} \times 100 \qquad (1)$$

Water samples

River water samples and wastewater effluents from textile industries (n = 3) were collected in light-protected polypropylene containers. Before the analysis of CV by the presented procedure, the water samples were passed through a 0.45 µm membrane filter. 15 mL of the sample was subjected to the procedure.

Calculation

The limit of detection (LOD) and limit of quantification (LOQ) were calculated using the following relations [34].

$$\mathrm{LOD} = \frac{3\,s}{S} \qquad (2)$$

$$\mathrm{LOQ} = \frac{10\,s}{S} \qquad (3)$$

where s is the standard deviation of 5 independent measurements of the blank sample while S is the slope of the calibration line after the extraction procedure.

RESULTS AND DISCUSSION

Optimization of the extraction procedure

The main factors needed for the extraction procedure, *i.e.*, pH, amount of MNPs, concentration of Triton X-114, temperature, and type of eluent, were optimized using the univariate method.

The impact of pH on the extraction of CV by the suggested procedure was investigated in the range of 4.0–8.0 by using the suitable buffer as mentioned in the experimental section. The results in Figure 1 imply that CV has maximal and quantitative extraction when pH is ≥ 5.0. As described in a previous study, this pH is higher than the point of zero charge (pH_{PZC}) of Fe_3O_4 ($pH_{PZC} = 2.1$) [35], thus its surface is negative, and the cationic molecules are adsorbed more effectively on the surface. Therefore, before processing CV-containing samples, the pH was adjusted to 5.0 by adding acetate buffer (1.0 mL, pH 5.0).

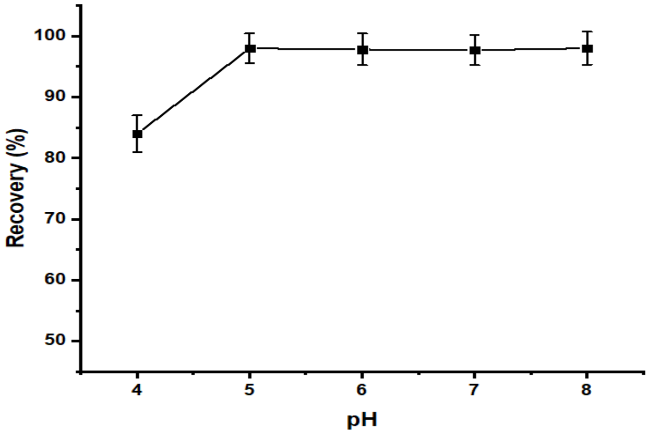

Figure 1. Effect of pH on the extraction of CV

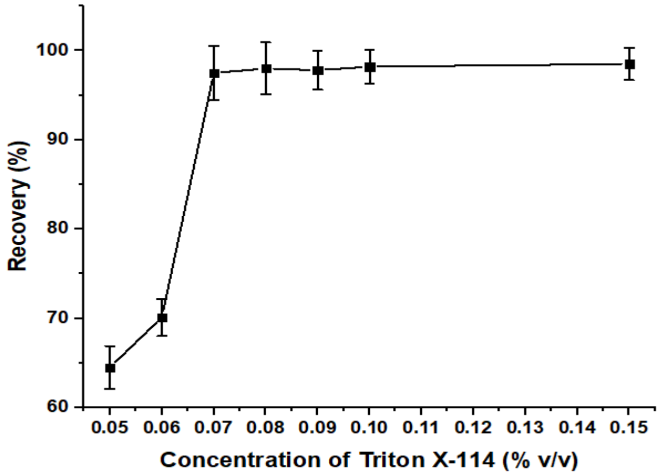

Figure 2. Influence of Triton X-114 on the extraction of CV.

Triton X-114 was used as an extraction solvent in this study owing to its commercial accessibility in pure form, low toxicological characteristics, and low cost. Furthermore, Triton X-114 has an adequate cloud temperature range (25-30 °C) making it suitable for the extraction of a wide range of analytes [36]. The effect of Triton X-114 on the extraction procedure was shown in Figure 2. As presented, the extraction increased by increasing the concentration of Triton X-114 and reached a maximum limit at 0.07 v/v% of Triton X-114. These findings which agree with other studies may be associated with micelle production; as the surfactant concentration increases, more micelles form in the solution, and more analytes shift to the micellar system [37, 38]. Once the micelles are extracted from the aqueous solution, the target analyte is also enriched by phase separation. Therefore, 0.07 v/v% of Triton X-114 was selected as the optimum level.

The effect of MNPs dose was presented in Figure 3. The results illustrate that when the quantity of MNPs is more than 4 mg, the extraction of CV remains nearly constant. As a result, the optimal adsorbent quantity for this process was 4 mg of MNPs. This amount is lower than amounts of applied adsorbents reported in similar previous studies in the literatures [28, 39, 40]. These results highlights the effectiveness of Fe_3O_4 for adsorption of CV from aqueous solutions.

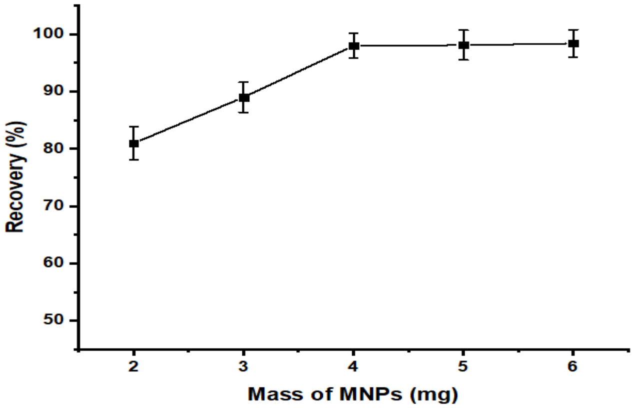

Figure 3. Effect of amount of MNPs on the extraction of CV.

Temperature is a vital variable for CPE since phase separation happens only if the temperature exceeds the cloud point temperature of the employed surfactant [41]. The consequences of equilibration temperature and time on the extraction of CV were examined and the results were summarized in Figure 4. As shown, the maximum extraction was achieved when the solution was equilibrated at 45 °C for 20 min. The extraction recovery improved as the temperature increased since the nonionic surfactant became more hydrophobic. As the temperature increased the surfactant's dehydration improved, contributing to micelle repulsion and aggregation [42]. Therefore, incubation at 45 °C for 20 min was chosen as the optimal equilibrium temperature and time, respectively.

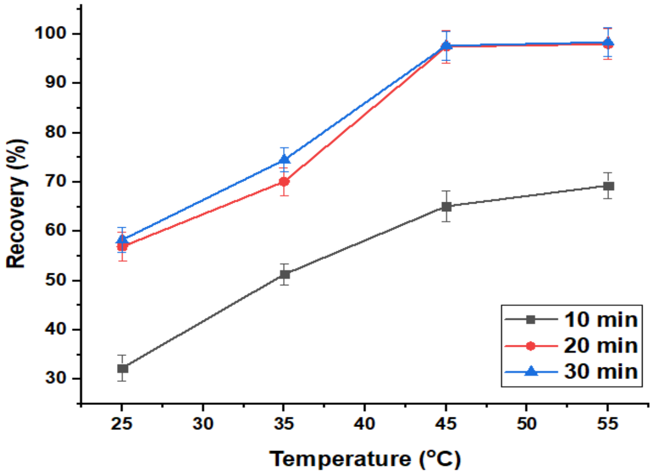

Figure 4. Effect of incubation temperature and time on the extraction of CV.

To release CV form MNPs that were enriched in the surfactant phase, several eluents (ethanol, 5:1 ethanol/HNO_3 mixture, acetone, and 0.1 mol L^{-1} NaOH) were investigated. According to the results presented in Figure 5, the ethanol/HNO_3 mixture (5:1 v/v) led to the maximum recovery. The effect of varied volumes of this solvent in the range of 250-500 µL was tested. The findings revealed that the recovery increased until it ultimately reached a plateau at 350 µL. Eluent levels exceeding 350 µL did not substantially enhance the extraction efficiency. Therefore, 350 µL of a 5:1 v/v ethanol/HNO_3 mixture was chosen as the eluent for the remaining assays. Considering that the sample size is 15.0 mL, the preconcentration factor (PF) for this is 42.9.

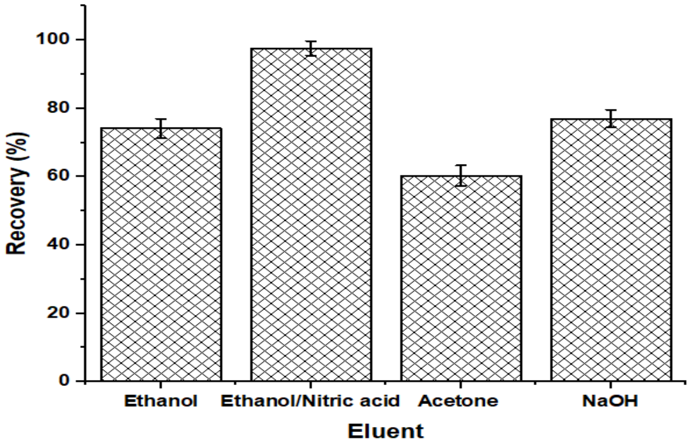

Figure 5. Effect of eluent type on the recovery of CV.

Selectivity

The effect of potential interfering species on the extraction of CV was studied by applying the present procedure to a binary mixture containing 100 µg L^{-1} of CV and different amounts of the concomitant species. According to its definition, the tolerance limit is the highest concentration at which the extraction recovery changes by less than 5% [43]. The findings in Table 2 show that the most common species present in real samples did not interfere with the extraction of CV indicating the selectivity of the procedure.

Table 2. Effect of concomitant species on the recovery of CV (n = 3).

Concomitant species	Tolerance limit (mg L^{-1})
Na^+, K^+, NH_4^+, Cl^-, NO_3^-, SO_4^{2-}	1000.0
Mg^{2+}, Ca^{2+}, Cd^{2+}, Pb^{2+}, Ni^{2+}, Cu^{2+}, Zn^{2+}, Al^{3+}, Fe^{3+}	500.0
Allura red, Rhodamine B	25.0
Brilliant green, eosin	5.0
Methylene blue	1.0

Analytical performance and comparison with other procedures

The analytical characteristics of the approach were assessed in the basics of linear range, limit of detection (LOD), and limit of quantitation (LOQ), under the optimum conditions. The procedure exhibits linearity in the range of 1.5–250.0 µg L^{-1}. The LOD and LOQ were calculated as 0.5 and 1.5 µg L^{-1} by applying Eq. (2) and Eq (3), respectively. For five duplicate analyses of CV at a concentration of 100 µg L^{-1}, the intra-day and inter-day relative standard deviations (RSD) were 2.1 and 1.7%, respectively.

A comparison between the current procedure and other previous approaches for the extraction of CV presented in Table 3. As we see, our procedure shows lower a LOD and a higher preconcentration factor (PF) than those of the other procedures.

Table 3. Comparison of analytical features with other extraction procedures for CV.

Extraction procedure	Linearity (µg L^{-1})	LOD (µg L^{-1})	LOQ (µg L^{-1})	PF	RSD (%)	Ref.
Cloud point extraction	2.0 – 400.0	0.7	2.0	25.0	-	[44]
Cloud point extraction	100.0 – 800.0	5.1	-	-	4.5	[45]
Magnetic-nanoparticle-based dispersive solid phase extraction	100.0 – 600.0	25.0	-	-	3.9 – 5.9	[46]
Solid phase extraction using SBA-3	Up to 200.0	1.3	-	200.0	1.3	[47]
Solid phase extraction using a rotating disk coated with polydimethylsiloxane	5.0 – 225.0	1.8	5.4	-	6.2 – 8.4	[48]
Cloud point extraction combined with the adsorption onto magnetic nanoparticles	1.5 – 250.0	0.5	1.5	42.9	1.7 – 2.1	This work

Application to wastewater samples

To validate the procedure, water samples were analyzed for CV using the optimized procedure. The accuracy was estimated by calculating the recovery of the target analyte after adding a known amount of CV to the sample. As listed in Table 4, there was substantial agreement between the added and measured CV concentrations. The recovery limits computed from the spiking standards ranged from 97.0 to 101.2% and RSD less than 2.2%. These findings demonstrated that the presented procedure could be utilized to separate and analyze CVs successfully.

Table 4. Analysis of CV in wastewater samples.

Sample	Added (µg L^{-1})	Measured (µg L^{-1})	Recovery (%)	RSD (%)
River water (The Nile River)	0.0	Not detected	-	-
	10.0	9.8±0.21	98.0	2.1
	25.0	25.3±0.37	101.2	1.5
Wastewater (textile effluents)	0.0	2.3±0.04	-	1.7
	10.0	12.0±0.26	97.0	2.2
	25.0	26.9±0.41	98.4	1.5

CONCLUSION

The findings of the work show that cloud point extraction, along with adsorption onto magnetic nanoparticles, is an effective procedure for the separation and determination of crystal violet. Because the MNPs were well dispersed in the aqueous phase, there is no need for centrifugation to separate the phases. Moreover, the technique is quite straightforward and inexpensive. This approach is precise and selective, with high recovery (97.0-101.2) and low detection and quantification limits (0.5 and 1.5, respectively). This simple approach could be used to detect trace quantities of crystal violet in water samples.

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Competing interests

The authors declare that they have no known competing financial interests or personal relationships that could have appeared to influence the work reported in this paper.

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Availability of data and materials

The datasets used or analyzed during the current study are available from the corresponding author upon reasonable request.

REFERENCES

1. Al-Tohamy, R.; Ali, S.S.; Li, F.; Okasha, K.M.; Mahmoud, Y.A.-G.;Elsamahy, T.; Jiao, H.; Fu, Y.; Sun, J. A critical review on the treatment of dye-containing wastewater: Ecotoxicological and health concerns of textile dyes and possible remediation approaches for environmental safety. *Ecotoxicol. Environ. Saf.* **2022**, 231, 113160.

2. Karatepe, A.; Akalin, C.; Soylak, M. Solid-phase extraction of some food dyes on sea sponge column and determination by UV–Vis spectrophotometer. *Desalin. Water Treat.* **2016**, 57, 25822-25829.
3. Ahmed, H.E.H.; Kori, A.H.; Gumus, Z.P.; Soylak, M. Supramolecular solvent-based liquid–liquid microextraction (SUPRAS-LLME) of Sudan dyes from food and water samples with HPLC. *Microchem. J.* **2024**, 201, 110682.
4. Mirza, A.; Ahmad, R. An efficient sequestration of toxic crystal violet dye from aqueous solution by Alginate/Pectin nanocomposite: a novel and ecofriendly adsorbent. *Groundw. Sustain. Dev.* **2020**, 11, 100373.
5. Berradi, M.; Hsissou, R.; Khudhair, M.; Assouag, M.; Cherkaoui, O.; El Bachiri, A.; El Harfi, A. Textile finishing dyes and their impact on aquatic environs. *Heliyon* **2019**, 5, e02711.
6. Ozalp, O.; Soylak, M. Microextraction methods for the separation-preconcentration and determination of food dyes: A minireview. *Anal. Lett.* **2023**, 56, 2473-2490.
7. Gebeyehu, B.T.; Tadesse, M.G.; Tedla, A. Simultaneous removal of methylene blue dye and waterborne pathogens from wastewater using extract of Embelia schimperi fruits. *Bull. Chem. Soc. Ethiop.* **2024**, 38, 281-296.
8. Thao, T.T.P.; Nguyen-Thi, M.-L.; Chung, N.D.; Ooi, C.W.; Park, S.-M.; Lan, T.T.; Quang, H.T.; Khoo, K.S.; Show, P.L.; Huy, N.D. Microbial biodegradation of recalcitrant synthetic dyes from textile-enriched wastewater by Fusarium oxysporum. *Chemosphere* **2023**, 325, 138392.
9. Benkhaya, S.; Lgaz, H.; Tang, H.; Altaee, A.; Haida, S.; Vatanpour, V.; Xiao, Y. Investigating the effects of polypropylene-TiO_2 loading on the performance of polysulfone/polyetherimide ultrafiltration membranes for azo dye removal: Experimental and molecular dynamics simulation. *J. Water Process Eng.* **2023**, 56, 104317.
10. Alkhouzaam, A.; Khraisheh, M. Towards sustainable management of end-of-life membranes: Novel transformation of end of life (EoL) reverse osmosis membranes for efficient dye/salt separation. *Desalin. Water Treat.* **2024**, 571, 117104.
11. Lanzetta, A.; Papirio, S.; Oliva, A.; Cesaro, A.; Pucci, L.; Capasso, E.M.; Esposito, G.; Pirozzi, F. Ozonation processes for color removal from urban and leather tanning wastewater. *Water* **2023**, 15, 2362.
12. Mortada, W.I.; Nabieh, K.A.; Abdelghany, A.M. Efficient and low-cost surfactant-assisted solid phase extraction procedure for removal of methylene blue using natural dolomite. *Water Air Soil Pollut.* **2023**, 234, 363.
13. Guan, X.; Zhang, B.; Li, D.; Ren, J.; Zhu, Y.; Han, Q.; Rao, P. *Adsorption-Based Filtration Membranes for Wastewater Treatment, in Adsorption Through Advanced Nanoscale Materials*. Elsevier: Online Publication; **2023**, pp. 259-285.
14. Raji, Y.; Nadi, A.; Mechnou, I.; Saadouni, M.; Cherkaoui, O.; Zyade, S. High adsorption capacities of crystal violet dye by low-cost activated carbon prepared from Moroccan *Moringa oleifera* wastes: Characterization, adsorption and mechanism study. *Diam. Relat. Mater.* **2023**, 135, 109834.
15. Ahmed, M.; Nasar, A. Magnetized bentonite clay particles: An eco-friendly and economical adsorbent for efficient remediation of crystal violet dye-polluted water. *Arab. J. Sci. Eng.* **2024**, 49, 801-813.
16. Rajamohan, R.; Lee, Y.R. Microwave-assisted synthesis of copper oxide nanoparticles by apple peel extract and efficient catalytic reduction on methylene blue and crystal violet. *J. Mol. Struct.* **2023**, 1276, 134803.
17. Foroutan, R.; Peighambardoust, S.J.; Ghojavand, S.; Foroughi, M.; Ahmadi, A.; Bahador, F.; Ramavandi, B. Development of a magnetic orange seed/Fe_3O_4 composite for the removal of methylene blue and crystal violet from aqueous media. *Biomass Convers. Biorefin.* **2024**, 14, 25685-25700.

18. Bhaduri, B.; Dixit, A.K.; Tripathi, K.M. *Magnetic Nanoparticles: Application in the Removal of Next-Generation Pollutants from Wastewater* in *New Trends in Emerging Environmental Contaminants. Energy, Environment, and Sustainability*, Singh, P.; Agarwal, S.; Gupta, A.K.; Maliyekkal, S.M. (Eds), Springer: Singapore; **2022**.
19. Guo, Y.; Chen, B.; Zhao, Y.; Yang, T. Fabrication of the magnetic mesoporous silica Fe-MCM-41-A as efficient adsorbent: Performance, kinetics and mechanism. *Sci. Rep*. **2021**, 11, 2612.
20. Li, Y.; Yang, C.-X.; Yan, X.-P. Controllable preparation of core–shell magnetic covalent-organic framework nanospheres for efficient adsorption and removal of bisphenols in aqueous solution. *Chem. Commun.* **2017**, 53, 2511-2514.
21. Verma, C.; Verma, D.K.; Berdimurodov, E.; Barsoum, I.; Alfantazi, A.; Hussain, C.M. Green magnetic nanoparticles: A comprehensive review of recent progress in biomedical and environmental applications. *J. Mater. Sci*. **2024**, 59, 325-358.
22. Płotka-Wasylka, J.; Szczepańska, N.; De La Guardia, M.; Namieśnik, J. Miniaturized solid-phase extraction techniques. *TrAC Trends Anal. Chem.* **2015**, 73, 19-38.
23. Liggieri, L.; Miller, R. Relaxation of surfactants adsorption layers at liquid interfaces. *Curr. Opin. Colloid Interface Sci.* **2010**, 15, 256-263.
24. Osaghi, B.; Safa, F. Cloud point extraction in presence of multi-walled carbon nanotubes for removal of direct green 26 from aqueous solutions: optimization by box-behnken design and desirability functions. *J. Anal. Chem*. **2020**, 75, 802-811.
25. Mortada, W.I.; Ali, A.-A.Z.; Hassanien, M.M. Mixed micelle-mediated extraction of alizarin red S complexes of Zr(IV) and Hf(IV) ions prior to their determination by inductively coupled plasma-optical emission spectrometry. *Anal. Methods* **2013**, 5, 5234-5240.
26. Abou El-Reash, Y.G.; Tantawy, H.A.; Abdel-Latif, E.; Mortada, W.I. Micelle-mediated extraction for speciation and spectrophotometric determination of iron using a newly synthesized salicylic acid derivative. *Microchem. J*. **2020**, 158, 105280.
27. Azooz, E.A.; Shabaa, G.J.; Al-Muhanna, E.H.B.; Al-Mulla, E.A.J.; Mortada, W.I. Displacement cloud point extraction procedure for preconcentration of iron (III) in water and fruit samples prior to spectrophotometric determination. *Bull. Chem. Soc. Ethiop*. **2023**, 37, 1-10.
28. Safa, F.; Osaghi, B. Adsorption onto MWCNTs coupled with cloud point extraction for dye removal from aqueous solutions: optimization by experimental design. *Comb. Chem. High Throughput Screen.* **2021**, 24, 246-258.
29. Abdelwahed, F.T.; Mortada, W.I.; El-Defrawy, M.M.; Eltabey, R.M. Lead extraction from food samples by combined cloud point-micro solid phase extraction. *J. Food Compos. Anal.* **2024**, 129, 106119.
30. Nekouei, F.; Kargarzadeh, H.; Nekouei, S.; Keshtpour, F.; Makhlouf, A.S.H. Efficient method for determination of methylene blue dye in water samples based on a combined dispersive solid phase and cloud point extraction using $Cu(OH)_2$ nanoflakes: Central composite design optimization. *Anal. Bioanal. Chem.* **2017**, 409, 1079-1092.
31. Pirdadeh-Beiranvand, M.; Afkhami, A.; Madrakian, T. Cloud point-magnetic dispersive solid phase extraction for the spectrofluorometric determination of citalopram. *J. Mol. Liq.* **2017**, 241, 43-48.
32. Khani, R.; Irani, M. Mixed magnetic dispersive micro-solid phase-cloud point extraction of Sunset Yellow in food and pharmaceutical samples. *ChemistrySelect* **2021**, 6, 273-278.
33. Mehdi, Z.S.; Alshamkhawy, S.A.R.A. A univariate optimization strategy for pre-concentration of cobalt(II) in various matrixes by a DLLME before analysis using FAAS. *Indones. J. Chem.* **2024**, 24, 403-414.
34. Ershadi, S.; Shayanfar, A. Are LOD and LOQ reliable parameters for sensitivity evaluation of spectroscopic methods? *J. AOAC Int.* **2018**, 101, 1212-1213.

35. Al-Salihi, S.; Bayati, M.; Jasim, A.M.; Fidalgo, M.M.; Xing, Y. Magnetic mesoporous TiO_2/Fe_3O_4 nanocomposite adsorbent for removal of sulfamethazine from water. *Environ. Adv.* **2022**, 9, 100283.
36. Snigur, D.; Azooz, E.A.; Zhukovetska, O.; Guzenko, O.; Mortada, W.I. Recent innovations in cloud point extraction towards a more efficient and environmentally friendly procedure. *TrAC Trends Anal. Chem*. **2023**, 164, 117113.
37. Ji, Y.; Wu, L.; Lv, R.; Wang, H.; Song, S.; Cao, M. Facile cloud point extraction for the separation and determination of phenolic acids from dandelion. *ACS Omega* **2021**, 6, 13508-13515.
38. Mortada, W.I.; El-Defrawy, M.M.; Erfan, E.; El-Asmy, H.A. Cloud point extraction coupled with back-extraction for speciation of inorganic vanadium in water and determination of total vanadium in food samples by ICP-OES. *J. Food Compos. Anal*. **2022**, 108, 104445.
39. Nekouei, S.; Nekouei, F.; Tyagi, I.; Agarwal, S.; Gupta, V.K. Mixed cloud point/solid phase extraction of lead(II) and cadmium(II) in water samples using modified-ZnO nanopowders. *Process Saf. Environ. Prot.* **2016**, 99, 175-185.
40. Gao, N.; Wu, H.; Chang, Y.; Guo, X.; Zhang, L.; Du, L.; Fu, Y. Mixed micelle cloud point-magnetic dispersive μ-solid phase extraction of doxazosin and alfuzosin. *Spectrochim. Acta A Mol. Biomol. Spectrosc.* **2015**, 134, 10-16.
41. Szymczyk, K.; Taraba, A. Aggregation behavior of Triton X-114 and Tween 80 at various temperatures and concentrations studied by density and viscosity measurements. *J. Therm. Anal. Calorim.* **2016**, 126, 315-326.
42. Arkhipov, V.P.; Arkhipov, R.; Filippov, A. Micelles of oxyethylated isononylphenols in aqueous solutions and hydrophilic-lipophilic balance. *ACS omega* **2020**, 5, 28224-28232.
43. Khalifa, M.E.; Mortada, W.I.; El-Defrawy, M.M.; Awad, A.A. Selective separation of gadolinium from a series of f-block elements by cloud point extraction and its application for analysis of real samples. *Microchem. J.* **2019**, 151, 104214.
44. Bisgin, A.; Narİn, İ.; Ucan, M.; Soylak, M. A new cloud point extraction procedure for determination of trace amount crystal violet in wastewater by UV-VIS. Spectrometry. *Oxid. Commun.* 2015, 38, 232-240.
45. Ghasemi, E.; Kaykhaii, M. Application of micro-cloud point extraction for spectrophotometric determination of malachite green, crystal violet and rhodamine B in aqueous samples. *Spectrochim. Acta A Mol. Biomol. Spectrosc.* **2016**, 164, 93-97.
46. Shojaei, S.; Rahmani, M.; Khajeh, M.; Abbasian, A.R. Magnetic-nanoparticle-based dispersive micro-solid phase extraction for the determination of crystal violet in environmental water samples. *ChemistrySelect* **2021**, 6, 4782-4790.
47. Azarkohan, A.; Shemirani, F.; Alvand, M. Fast analysis of water samples for trace amount of crystal violet dye based on solid phase extraction using nanoporous SBA-3 prior to determination by fiber optic-linear array detection spectrophotometry. *J. Chem*. **2013**, 2013, 530843.
48. Manzo, V.; Navarro, O.; Honda, L.; Sánchez, K.; Toral, M.I.; Richter, P. Determination of crystal violet in water by direct solid phase spectrophotometry after rotating disk sorptive extraction. *Talanta* **2013**, 106, 305-308.