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Fast and green separation of malachite green in water samples by micro-dispersion scanometry method without heating, cooling and organic solvents at room temperature



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ABSTRACT

In the present work, novel, easy, rapid, green, and, economical technique, micro-dispersion scanometry (MDS) is presented for the first time and employed using suspended Ni(OH)2 nanopowder in the micellar medium for the determination of slight amounts of malachite green chloride. In the meanwhile, we introduced a new simple method for the synthesis of Ni(OH)₂ nanopowder followed by characterization via various methods such as scanning electron microscopy (SEM), X-ray diffraction (XRD) and, Brunauer, Emmett and Teller (BET). The process of scanning (detecting) was done on the cells including the sample solution via a usual flatbed-scanner. Then, for analyzing the color of the cells, a software system designed in Visual Basic (VB 6), to R (red), G (green), and B (blue) values was applied. To build the cells, some holes were created in the plexiglas sheet. The impact of experimental variables namely pH, weight of sorbent, volume of 4% Triton X-114, eluting solution, and sample volume have been investigated and optimized in multivariate method using design Expert 7.0 software for statistical data analysis. A contrast done between the proposed and traditional UV-vis spectrophotometry methods revealed the comparable tendency in both methods Calibration curves were linear in the range of 0.37–110 and 0.45–110 $\mu g\,L^{-1}$ for scanometry and UV–vis methods, respectively. The detection limits were 0.060 and 0.068 μ g L⁻¹ for scanometry and UV-vis methods, respectively. The capacity of Ni(OH)₂ nanopowder for malachite green was $80 \,\mathrm{mg \, g^{-1}}$.

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1. Introduction

Industrial wastewater evacuated in nature such as the textile, leather tanning, paper production, food technology, hair colorings, etc., are commonly contaminated by dyes. Malachite green is one of the most usual dyes applied for the coloring of cotton, silk, paper, leather and also in making paints and printing inks. majority of the dyes, comprising malachite green, are noxious and have to be eliminated before evacuating into rivers (Hameed and El-Khaiary, 2008).

Nowadays, there are various methods for removal of dyes and other pollutants from water samples including adsorption (Asfaram et al., 2015; Balarak et al., 2015; Gupta et al., 2011, 2013, 2012b; Jain et al., 2003; Mittal et al., 2009a,b, 2010a, 2010b; Mohammadi et al., 2011; Nekouei et al., 2016b,c,d, 2015), cloud point extraction

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(Abedi and Nekouei, 2011; Mousavi and Nekouei, 2011), biodegradation (Nekouei and Nekouei, 2017c), physicochemical oxidation processes (Karthikeyan et al., 2012; Nekouei et al., 2017b), and photocatalyst (Gupta et al., 2012a; Nekouei and Nekouei, 2017a,b; Saleh and Gupta, 2012; Saravanan et al., 2014, 2015, 2013a,b,c, 2016), which are repeatedly reported in the manuscripts. This new approach is established upon simultaneous micro-cloud point and solid phase extraction detected by scanometry method. In regular cloud point extraction (CPE), micellar solution of non-ionic surfactant is formed by heating and the hydrophobic analyte is transferred from aqueous solution to micellar phase. Heating (usually is water bath) is the key factor of forming the micellar phase which is named cloudy solution. This step is one of the drawbacks of cloud point extraction due to time consuming that varies from 20 min to 4 h. In light of the time required for the centrifuging and application of cold bath to condensation of the enriched surfactant phase, the stage of CPE is time consuming, making it as a long methodology. To modify this method as a fast method, we applied a modification on the procedure by means of salting out rather than heating step (Ghasemi and Kaykhaii, 2015, 2016). As in this methodology the small quantity of the surfactant rich phase achieved; thus, the extraction has some advantages including being simple, inexpensive, highly effective, fast, and with lower toxicity as compared to the extractions that use organic solvents (Nekouei et al., 2016e). The base of this work is established upon micro-cloud point extraction-solid phase extraction-scanometry which the nanopowder is involved in micro-cloud point extraction (MCPE) technique, resulting dispersion of nanoparticles in the micellar medium detected by scanometry method (micro-dispersion-scanometry). The phase separation phenomenon has been used for the extraction and separation of malachite green from water samples.

Mainly, nanostructured materials have potential preferences for dye removal applications owing to their large surface area and a large amount of atoms attainable for physical and chemical interactions (Ran et al., 2012). Nickel hydroxide compounds stimulated a large interest in industrial and technical applications, for example as the active materials in electrochemical cycling and as a photocatalyst to remove organic dyes. Furthermore, the feasibility of Ni(OH)₂ and NiO nanomaterials for their application in water treatment is also reported and evaluated (Song and Gao, 2008).

In this work, scanometry was applied as detector. In the scanometric method, first the solution is scanned with a flatbed scanner, and afterwards, the image is under investigation of an image analyzer software. In the image analyzer software the color spots are analyzed to red, green and blue color values. In contrast to spectrophotometry, in scanometry the solution does not need to be transparent, since the light reflection is determined, and the light radiance does not pass through the solution. In the proposed work, the λ_{max} is not of importance since in this method we are dealing only with color intensity (Abbaspour et al., 2009). In This process the plexiglas cells that contain colored solutions are scanned and finally the R (Red) G (green) B (blue) model is applied in color monitoring. Scanometry has some advantages including ease of use (handheld scanner and PC), fast scanning, inexpensiveness, portable systems and easy immobilization of reactants, lack of necessity for determining the $\lambda_{\text{max}},$ exact results of experiments, small time of response, very low interference, and the scope for performing various simultaneous tests, utilizing nontransparent solutions and study of the reflective characteristics of the surface (Shokrollahi et al., 2015). However, the disadvantages associated with spectrophotometry are the high price of the implement and its maintaining, high interfering and short dynamic extent (Shokrollahi and Roozestan, 2013).

Surface enhance methodology (RSM) is a collection of statistical and mathematical methods that are useful for modeling and analyzing engineering problems. The main objective of RSM is to optimize the response surface that is influenced by various process parameters. RSM also quantifies the relationship between the controllable input parameters and the obtained response surfaces (Fakhri et al., 2016; Nekouei et al., 2016a, 2017c).

In the present study, we have firstly synthesized $\rm Ni(OH)_2$ nanopowder with a new technique. In this study, we report a new technique

for the first time named dispersion-scanometry by Ni(OH)₂ nanopowdres for extraction of malachite green chloride. It is for the first time that dispersive method is done at room temperature without cooling, heating, and organic solvents which is very time effecting and green. Moreover, it is for the first time dispersive method is done by sacanometry. The developed method was validated with traditional dispersion spectrophotometry. Finally, the RSM (central composite design (CCD)) was applied to design the effect of some functioning variables on the accomplishment of the malachite green extraction using design Expert 7.0 software.

2. Experimental

2.1. Instrumentation

A Metrohm pH-meter (model 691, Switzerland) was used in order to adjust the pH at desirable values. The cells are made using a sheet of plexiglas. CanoScan 5000F (Canon) scanner to scan the plexiglas sheet. The scanner resolution was adjusted on 300 dpi. The software was written in VB 6 media to convert the color pictures of the cells to RGB data. A 100–1000 μL Biohit proline pipettor was applied to inject the samples into the cells. Absorbance measurements were done using Jasco V-550, UV-vis spectrophotometer with 10 µL microcells. A centrifuge (Hettich, Universal 120, England) was applied to complete phase separation. A magnetic stirrer (Jeotech, Korea) was utilized for all steps in experiments. X-ray diffraction (XRD) measurements were conducted using a Bruker AXS-D8 Advance model system. Scanning electron microscopy (SEM) was performed on Zeiss Supra, model 55VP, Germany. A BET surface analyzer (Micromeritics ASAP 2000, USA) was used for surface area evaluation. Microwave oven (Samsung, Korea, 900 W, 2.45 GHz frequency) to dry the powder formed. A Pyris Diamond thermogravimetric apparatus to analyze the thermal behavior of nanomaterial. A Fourier transformed infrared spectrometer (FTIR Nicolet iS5 Thermoscientific, UK) in the wavelength range of 400–4000 cm⁻¹.

2.2. Standard solutions and reagents

Malachite green (Aldrich) (Fig. 1s) was utilized for the preparation of the stock solutions of Malachite green in desired concentrations. 4.0% (v/v) solution of Triton X-114 was provided by diluting 4 mL of surfactant to 100 mL by water. A stock solution of $0.08 \text{ mol L}^{-1} \text{ Na}_2 \text{SO}_4$ was acquired by dissolving appropriate amount of salt in 100 mL water. The working standard solutions were prepared by diluting stock standard solution. Britton–Robinson buffer solution in the pH range of 4–11 was applied to regulate pH.

2.3. Synthesis procedure of Ni(OH)₂

Nickel hydroxide was provided by dissolving 0.5 g of Nickel nitrate Ni(NO₃)₂·4H₂O (Aldrich) into 50 mL distilled water in a 100 mL flask. Afterwards, 10 mL of NaOH 3 M and 2 mL of 0.01 M CTAB were added slowly at 80 °C with constant stirring for 55 min at pH 8. Afterwards, the suspension was filtered and rinsed by a combination of distilled water and ethanol to remove impurities. Then the mixture was dried by microwave oven for 8 h before being characterized. After that the green powder was formed and characterized.

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