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NETCHEM Remote Access Laboratory Guide

Photocatalytic degradation of crystal violet color on TiO_2 as catalyst



In this exercise, you will:

- Measure a series of standard dye solution with different concentration
- Measure change of absorption color on catalyst with/without UV-radiation
- Perform chemical analysis on UV-Vis spectrophotometer
- Record the absorption of violet die
- Gain experience in UV-Vis usage

Background

In this experiment, we use Sol-gel method, the use of titanium(IV)-isopropoxide as a precursor, to synthesized a catalyst based on TiO_2 . pH of the solution was adjusted using 1 M NaOH solution. After the precipitation of titanium(IV)-isopropoxide, the experiment was carried out by filtration, than washing of the residue (with distilled water and isopropanol), and at the end drying in an oven at 100°C and calcination at 450°C in static conditions.

The experiment consists of two parts:

1. test of adsorption
2. photocatalytic decomposition of a pollutant model (the color of crystal violet)

Material

For this lab exercise, you will need the following material :

- TiO₂
- crystal violet,
- Destiled water

- Apparatus:
- Analytical Balance
- Standard courts (100 cm³, 200 cm³)
- Petri dish
- magnetic stirrer
- Centrifuge
- UV-lamp
- UV-Vis spectrophotometer

Procedure:

- I. Molar mass of crystal violet die is $M(C_{25}N_3H_{30}Cl)=407.979$ g/mol. At first prepare a standard solution of die concentration of 1 mmol/dm³, by measuring 0.0816 CV and dissoving it in standard court od 200 cm³.
- II. Calibration standards: Prepare a set of crystal violet die solutions different concentrations: 0.002 mmol/dm³, 0.004 mmol/dm³, 0.006, 0.008, 0.01 and 0.012 mmol/dm³.
- III. Record the spectrum of each of the 6 standards with water in reference cuvette. based on the measured absorbance draw calibration curve as a absorbance versus concentration for each compound.
- IV. Adsorption test involves the monitoring of the dye adsorption on the catalyst, in the dark, without the influence of UV radiation. Measured 50 g of TiO₂ which has been previously synthesized by sol-gel process and then calcinated at 450°C.
- V. In Petri dishes pour masured mass of catalyst and overflow with 10 cm³ crystal violet die concentration of 0.01 mmol/dm³.
- VI. Follow a potential color adsorption on a heterogeneous catalyst, for 1-2 hours, measuring the change in absorbance at wavelength of 590 nm using a spectrophotometer.
- VII. After have been found that the adsorption process is completed, the sample is exposed to the effects of UV-rays of wavelength 366 nm. The solution was stirred at a magnetic stirrer during the adsorption test and during the dye adsorption photocatalytic treatment.



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Procedure:

During the exhibition of color to UV-radiation, absorption measured every hour. This procedure requires the measurement of an aliquot of the treated solution, centrifuge it in order to separate the solution from the catalyst powder

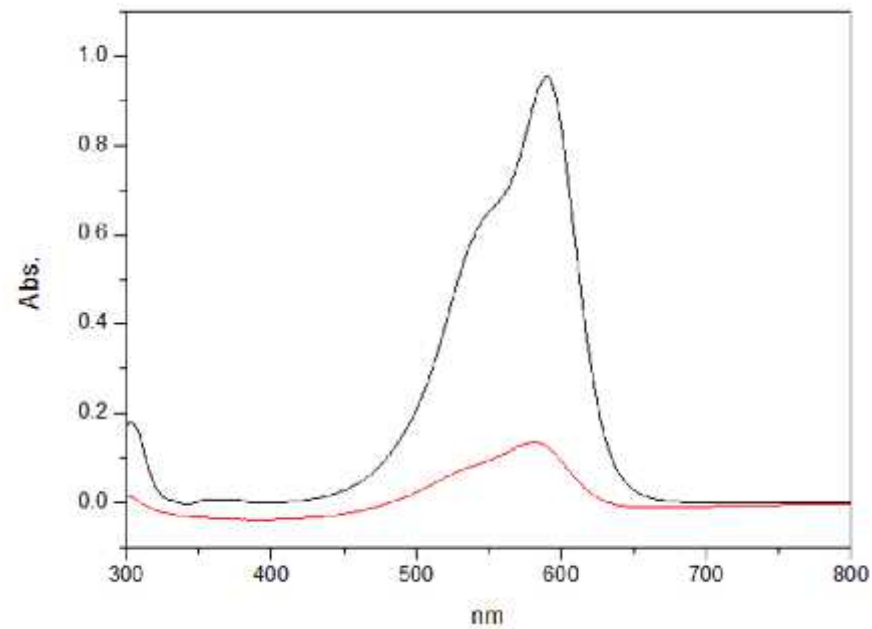
After each measurement, an aliquot of the solution quantitatively returned to the working solution.

Monitor the color degradation over time, the solution is decoloured.
Based on the results draw the curve color decolorization with time.



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Absorption spectrum of violet die before and after exposure to UV radiation





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Table 1. Dependence of absorbance versus concentration during time

Time (h)	Concentration of crystal violet die (mmol/dm ³)	Absorbance
0	0.01	0.970
1	0.002	0.163
2	0.001	0.123



DESCRIPTION OF REMOTE ACCESS	
1. NETCHEM COMMUNICATION SIDES	
(NOTE: NETCHEM Communication is defined as event that involves all kinds of internet interactions (in real time and not in real time) between participants via devices (PCs, laptops, tablets and mobilephones))	
host side (NOTE: Host side of NETCHEM Communication is defined as PC who invites other users to join the session)	participant's PC in classroom
guest side (NOTE: Guest side of NETCHEM Communication is defined as PC who joins the invitation to session)	participant's PC in laboratory
2. COMMUNICATION SOFTWARE	
Team Viewer	Meeting: No
	Remote control: No
	Meeting and Remote control simultaneously: No
Skype	Call 1:1: No
	Conference Call: Yes
3. COMMUNICATION HARDWARE	
on host side	1 PC for each participant
on guest side	1 PC, 1 headsets with microphone, camera
4. INFORMATION EXCHANGE TYPE	
Educational (one side is dominantly receptive)	Yes
	Place of Educator participant: guest side
	Number of educator(s): 1
	Place of student participant: host side
Consultative (two sides are equal in giving-receiving information)	Number of student participant(s): 15
	Number of host side participant(s): No
	Number of guest side participant(s): No





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Remote Access Connection Instructions

What makes these labs different and unique from other classroom experiments is that we have incorporated a section in each activity to remotely characterize your samples from your classroom.

Request a remote lab session specifying information such as: the day, the time, and the instrument you are interested in using by visiting our web site:

<http://netchem.ac.rs/remote-access>

You will see the list of partners with the instruments provided to choose from.

You will be contacted by a Remote Access staff member to set up a test run to ensure you are set up properly and have the required infrastructure.

Send samples or verify the in-house sample you would like us to prepare and load for characterization.

Send your samples to the Remote Access center that you chose on your request.

There are two communications software packages, that will allow us to communicate instructions and answer questions during the session.

- TeamViewer: You can obtain a free download at:

<https://www.teamviewer.com/en/index.aspx>

- Skype



Remote Access Connection Instructions

You will need:

- a) Computer with administrator access to install plug-ins and software
- b) An internet connection
- c) Speakers
- d) Microphone
- e) Projector connected to the same computer
- f) Web browser (Firefox preferred)

During the test run you can refer to this guide to perform the following steps, but it's very important that you only proceed with these steps during your scheduled times. You may interfere with other remote sessions and potentially damage equipment if you log in at other times.

- a) Open and logon to your Zoom/Team-viewer account. You will be given the access code to enter at the time of your test and then again during the remote session.
 - If you are using the Zoom software, Remote Access staff will give you the access code.
 - If you are using the Team-viewer software, Remote Access staff will give you the ID & password.
- b) You should soon see the Remote Access desktop and at this point you can interact with the icons on the screen as if it were your desktop.
- c) Switch to full screen mode by selecting the maximize screen option in the top right corner of the screen.
- d) Upon completion of the session, move your mouse to the top right corner of the screen, and click on the X to disconnect the remote session. It will ask if you want to end the remote session. Click Yes.



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Author, Editor and Referee References

This remote access laboratory was created thanks to work done primarily at University of Niš.

Contributors to this material were: dr Emilija Pecev-Marinkovic

Refereeing of this material was done by: _____

Editing into NETCHEM Format and onto NETCHEM platform was completed by: _____



References and Supplemental Material

The NETCHEM platform was established at the University of Nis in 2016-2019 through the Erasmus Programme.

Please contact a NETCHEM representatives at your institution or visit our website for an expanded contact list.

The work included had been led by the NETCHEM staff at your institution.