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INTERACTION OF N,N´-SUBSTITUTED DIIMIDES OF BINAPHTHYLHEXACARBOXYLIC ACID WITH SODIUM DITHIONITE IN AQUEOUS-ALKALINE ACID SOLUTION

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Abstract. *The article presents the details about the interaction of a number of N,N´-substituted diimides of binaphthylhexacarboxylic acid (cubogenes) with sodium dithionite. The reaction takes place in the formation of perylene dyes in the technology of dyeing and printing of cotton fabrics. The authors found that there is a rapid formation of intermediate products - tetraanions of initial compounds due to the transfer of two electrons from dithionite anions in the initial stage of the reaction. Subsequently, these intermediates can be transformed into the initial dianions by oxidation either by air oxygen or by sulfite formed by dithionite at the first stage. Also, a parallel chemical stage of cyclisation with the formation of perylene derivatives is possible depending on the nature of substituents at nitrogen atoms in the initial diimide molecules. The occurrence of a reversible redox stage of tetraanion formation confirmed electrochemically on a carbon-titanium electrode. The elemental analysis of IR and electronic absorption spectroscopy show the formation of perylene derivatives. The authors proposed a general stoichiometric reaction mechanism for the interaction reactions of cubogens having different substituents at nitrogen atoms with sodium dithionite on the basis of the experimental data. The results are applicable in the practice of finishing textile materials with cubogenes and in liquid-phase preparation of thin-layer photoactive materials based on perylene derivatives.*

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Introduction

Derivatives of N,N´-diimides of 1,1´-binaphthyl-4,4´,5,5´,8,8´-hexacarboxylic acid (DBHCA), or cubogenes, have been used as dyes for cotton, linen, and viscose materials since the 1970s [1, 2]. Textile technology uses their ability to cyclise and form durable perylene dyes on fabrics under the action of ‘soft’ reducing agents such as sodium dithionite, thiourea dioxide, and rhongalite [3, 4].

Recently, N,N'-diimide derivatives of 3,4,9,10-perylenetetracarboxylic acid are of great interest as they have found applications in solar energy converters [5-9], biosensors [10-13], and laser technology [14-17]. A common method for their preparation is the condensation of perylenetetracarboxylic acid or its dianhydride with the corresponding amines in high-boiling organic solvents or in pressurised aqueous medium [18]. As a result, compounds insoluble in aqueous solutions and poorly soluble in organic solvents are formed. Obtaining N,N'-diimides derivatives of 3,4,9,10-perylenetetetracarboxylic acid (DPTCC) by cyclisation of cubogens are well soluble in aqueous-alkaline solutions and are a good alternative. It allows the synthesis of perylene derivatives on various kinds of fibrous, porous, smooth surfaces to form films of different thicknesses and structures. Thus, in [19] it was shown that the deposition of DPTCC derivatives on porous titanium oxide by reductive cyclisation of DBGCC derivatives in aqueous-alkaline solution makes it possible to obtain composites with photoactive properties.

A number of articles [3, 4, 20-22] have been devoted to the study of the stoichiometric mechanism of the reductive cyclisation reaction of these derivatives. However, the composition and amount of the reaction products formed depend both on the nature of substituents at nitrogen atoms in the composition of DBGCC molecules and on the nature of the sulfur-oxygen reducing agent (sodium dithionite, thiourea dioxide, sodium hydroxymethanesulfinate). Therefore, the yield of the cyclisation product - perylene derivative - is 93-97%; N,N′-(2,5-dimethylphenyl)diimide of 1,1′- binaphthyl-4,4′,5,5′,5′,8,8′-hexacarboxylic acid interacts with sodium hydroxymethanesulfinate [20]; the yield of the cyclisation product - perylene derivative - is 35-50% when interacting with sodium dithionite under the same conditions; thiourea dioxide is not higher than 2%. Moreover, the composition and amount of intermediate and final cyclisation products depend on the concentration of the reducing agent and the presence of air oxygen in the solution [4]. These sulphoxygen compounds reducing activity is possessed by the molecules themselves, and by intermediates different in nature and reactivity. They are formed during the decomposition of sulfoxylic acid anions, dithionite, sulfur dioxide radical anions [3, 23].

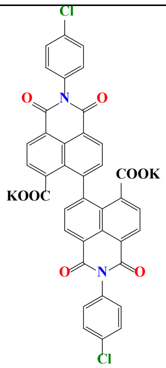
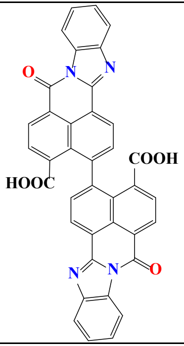
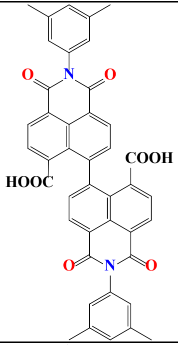
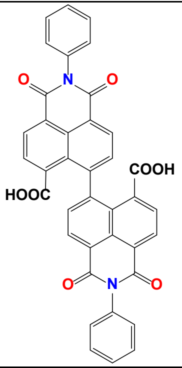
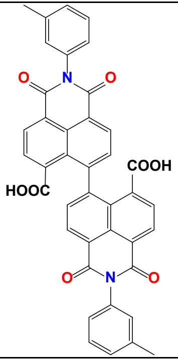
Indeed, an intermediate product is formed at the first stage of interaction reaction of DBGCC derivatives with sulfur-oxygen reducing agents. This product cannot be isolated from solution and fully identified. It is supposed to be either a dianion or an anion-radical particle of the initial cubogen molecule. Its further interaction with reducing particles causes the formation of cyclisation products either with cleavage of two carboxyl groups from the initial molecules of DBGCC derivatives (reaction with sodium hydroxymethanesulfinate) or without cleavage of carboxyl groups (reaction with thiourea dioxide).

The purpose of the present study is to reveal the nature of the intermediate compound formed during the primary stage of cubogenes reduction by sodium dithionite. However, its formation determines the further course of the reaction and the yield of cyclisation products.

Experimental part

Initial samples of cubogenes (Table 1) were obtained and purified at the State Scientific Centre NIOPIK (Moscow, Russia). We used sodium dithionite from ALBITESRL company (Italy) as a reducing agent with 89.2% of basic substance content.

Table 1. Structural formulas and names of the cubogens under study

№	Structural formula	Name
(1)		bis(Cl-Ph)Cub is the potassium salt of N,N‘- di(4-chlorophenyl)diimide of 1,1’-binaftyl-4,4‘,5,5’,8,8'-hexacarboxylic acid; M = 777.65 g/mol
(2)		bis(BZI)Cub is N,N‘-di(benzimidazole)diimide of 1,1’-binaphthyl-4,4‘,8,8’-dicarboxylic acid; M = 626.58 g/mol
(3)		bis(3,5-$(CH_3)_2$Ph)Cub is N,N‘-di(3,5-dimethylphenyl)diimide of 1,1’-binaftyl-4,4‘,5,5’,5‘,8,8’-hexacarboxylic acid; M = 688.69 g/mol
(4)		bis(Ph)Cub is N,N‘-di(phenyl)diimide of 1,1’-binaftyl-4,4‘,5,5’,8,8'-hexacarboxylic acid; M = 632.58 g/mol
(5)		bis(3-(CH_3)Ph)Cub is N,N‘-di(3-methylphenyl)diimide of 1,1’-binaftyl-4,4‘,5,5’,8,8'-hexacarboxylic acid; M = 660.64 g/mol

We recorded mass spectra on a Shimadzu Axima Confidence time-of-flight mass spectrometer (MALDI-TOF). We recorded infrared spectra on a TENSOR II FT-IR spectrometer (Bruker AXS Gmbh) and performed elemental analysis on a FLASH EA1112 Termo Quest carbon, hydrogen, nitrogen, sulphur and oxygen analyser with electronic microbalance. We obtained electronic absorption spectra using a LEKISS 2110 UV spectrophotometer (Finland) and quartz cuvettes.

We used a voltammetric analyser Ecotest-VA (Russia) with a three-electrode system to obtain cyclic voltammetric dependences. It consists of a rotating carbon steel working electrode, an auxiliary - impregnated - graphite electrode and a saturated silver chloride reference electrode EVL-1M4.

Methodology for the reaction of the interaction of cubogenes with sodium dithionite.

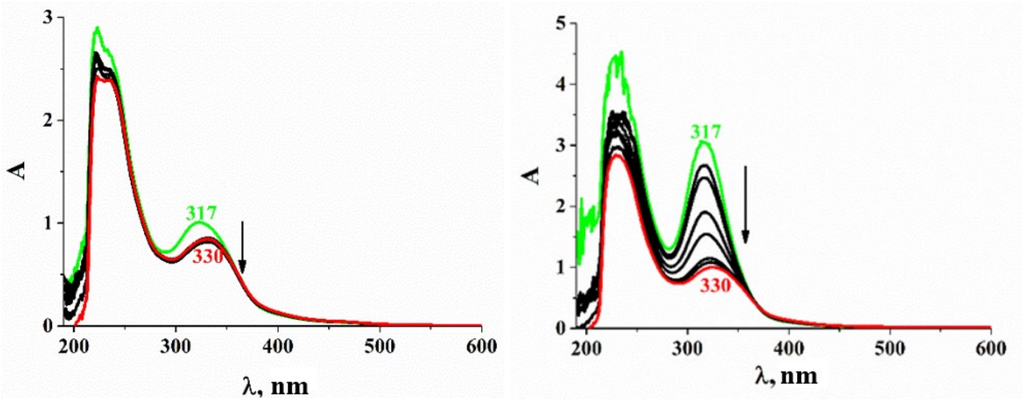
We dissolved cubogenic suspensions weighing approximately 0.003 g in 50 ml of 0.13 M sodium hydroxide solution. We dissolved sodium dithionite in a 50 ml measuring flask and brought to the mark with distilled water. We mixed the resulting solutions and poured them into the cuvette of the spectrophotometer if the reaction was performed under aerobic conditions. We blew argon through the prepared solutions before mixing them for 5-7 min under anaerobic conditions. We performed all experiments at a temperature equal to 300 K.

N,N'-di(3,5-dimethylphenyl)diimide-3,4,9,10-perylenetetracarboxylic acid. We filtered, dried, and analysed the red precipitate formed after completion of the cubogen bis(3,5-$(CH_3)_2$Ph)Cub reaction and holding the reaction mass in air. IR spectrum, ν, cm^{-1}: 1642 s (C=O), 1722 cp (C=O), 1665 s ($C-N_{imide}$), 792 s, 1260 s, 1460 s (perylene), 2800 sl ($-CH_3$). Electronic absorption spectrum (chloroform), λ_{max}, nm: 524, 564, 611. Mass-spectrum, m/z (I_{rel}, %) 600.5 (99). Found, %: C 79.80; H 4.53; N 4.42. $C_{40}H_{28}N_2O_4$. Calculated, %: C 80.00; H 4.66; N 4.51.

N,N'-di(benzimidazole)diimide-3,4,9,10-perylenetetracarboxylic acid. We filtered, dried,and analysed the violet-coloured precipitate formed after the cubogen bis(BZI)Cub reaction was completed and the reaction mass was kept air-dried. IR spectrum, ν, cm^{-1}: 800-750 s ($C-H_{benzimidazole}$), 1642 s (C=O), 1722 sr (C=O), 1665 s ($C-N_{imide}$), 792 s, 1260 s, 1460 s (perylene). Electronic absorption spectrum (aqueous-alkaline solution), λ_{max}, nm [lgε]: 330 [3.51], 421 [3.85]. Mass-spectrum, m/z (I_{rel}, %) 536.54 (99). Found, %: C 80.37; H 3.13; N 10.50. $C_{36}H_{16}N_4O_2$. Calculated, %: C 80.58; H 3.01; N 10.44.

Main body

Changes in the electronic absorption spectra are observed upon interaction of all the studied cubogens with sodium dithionite in aqueous-alkaline solution. A hypsochromic shift of the absorption band is observed in all cases in both aerobic and anaerobic atmospheres. It has a maximum at 330 nm; the appearance of a more intense band at 317 nm was on the example of the potassium salt of N,N'- di(4-chlorophenyl)diimide of 1,1'-binaftyl-4,4',5,5',8,8'-hexacarboxylic acid (Fig.1.).



(a) (b)

Fig. 1. Changes in the electronic absorption spectra of the bis(Cl-Ph)Cub interaction in aqueous alkaline solution (C_{NaOH} = 0.13 mol/l) at low (C_{red} = 3.68·10^{-4} mol/l) (a) and high (C_{red} = 1.84·10^{-3} mol/l) (b) concentrations of sodium dithionite. T = 306 K, $C_{cub.}$ = 3.9·10^{-5} mol/l. The green spectrum is the solution after mixing the reagents, the red spectrum is the solution after the reaction is complete.

The above changes in the cubogen spectra occur almost instantaneously when the reagents are mixed. Subsequently, the intensity of the spectral band with a maximum at 317 nm slowly decreases with time. Eventually, the absorption spectra are transformed into the initial spectra of cubogenes with a maximum at 330 nm for all studied cubogenes and 330 and 420 nm for bis(BZI)Cub. Meanwhile, the decrease in the intensity of the band with a maximum at 317 nm is faster in aerobic atmosphere. At the same time the decrease of the band intensity for all cubogens is slower in anaerobic atmosphere. The intensity of bis(3-(CH_3)Ph)Cub and bis(Ph)Cub in anaerobic atmosphere at high concentrations of sodium dithionite can remain unchanged for several days.

Absorption bands with maxima at 536 and 685 nm appear in the absorption spectra during the interaction of bis(BZI)Cub with sodium dithionite both in aerobic and anaerobic atmospheres at high concentrations. Moreover, an isobestic point is observed in the electronic spectra, which indicates that the reaction has a sequential stage of formation of one of the products (Fig. 2). Similar changes in the spectra were also observed for the cubogens bis(3,5-(CH_3)$_2$Ph)Cub and bis(Ph)Cub. However, the intensities of the bands in the long-wavelength part of the spectrum were insignificant.

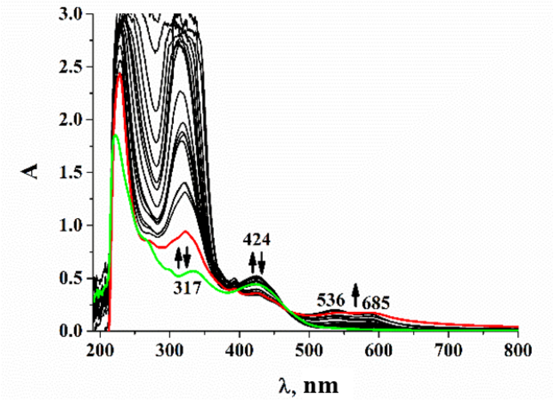


Fig. 2. Changes in electronic absorption spectra upon interaction of bis(BZI)Cub with sodium dithionite under aerobic conditions in a water-alkaline solution. C_{NaOH} = 0,13 mol/l; C_{reduct} = 1,84·10^{-3} mol/l; C_{cub} = 3,9·10^{-5} mol/l; T = 300 K. The green spectrum is the solution after mixing the reagents, the red spectrum is the solution after the reaction is complete.

Indeed, the action of reducing agents on DBGCC derivatives results in the cyclisation of the molecules to form perylene derivatives. Analysis results of the final products of bis(3,5-(CH_3)$_2$Ph)Cub and bis(BZI)Cub with dithionite are given in the experimental part of the study. According to them, the absorption bands with maxima at 536 and 685 nm are the products of cyclisation of these cubogens - N,N'-diimides of perylenetetracarboxylic acids with appropriate substituents. These compounds are present as dianions in aqueous alkaline solution.

The absorption bands in the electronic spectra with maxima at 317 nm for all the studied cubogenes can be attributed to the corresponding tetraanions of the initial compounds. They are formed due to the addition of two electrons from the reducing agent - the dithionite anion -$S_2O_4^{2-}$. Our paper [22] shows the formation of bis(BZI)Cub tetraanions during the electrochemical reduction of the initial cubogen in an aqueous-alkaline solution on a carbon-sitalloy electrode.

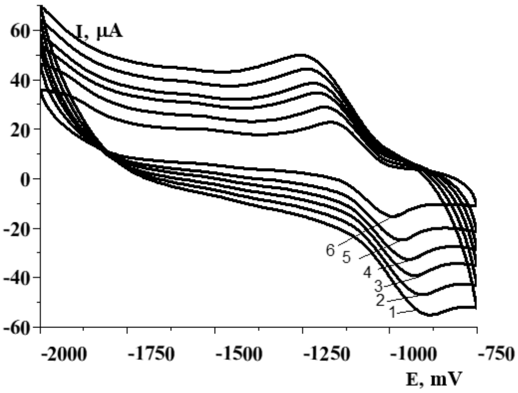


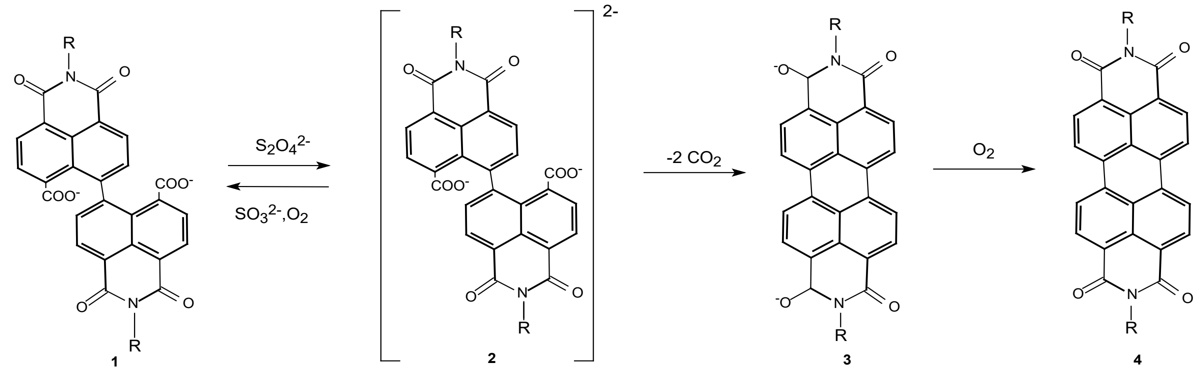
Fig. 3. [22] Effect of potential sweep speed (scan rate) on the voltammetric curves. *v*, mV/s: 1-20; 2-40; 3-60; 4-80; 5-100; 6-120.

Fig. 3 shows the voltampere dependences. There are two peaks corresponding to the cathodic and anodic processes of cubogen conversion. It indicates the reversibility of the electrode reaction.

The analysis of the above dependences showed participation of two electrons in the reversible electrode process. At the same time, sodium dithionite is also a two-electron reducing agent.

Based on the above data, a general stoichiometric reaction mechanism for the interaction of N,N´-diimides of 1,1´-binaphthyl-4,4´,5,5´,8,8´-hexacarboxylic acid (DBGCC) with sodium dithionite can be assumed (Scheme 1). Reversible reduction of initial molecules with formation of tetraanions (2) occurs in the primary stage of the reaction of interaction of sodium dithionite with cubogenes (1). Further, the chemical stage of cyclisation with detachment of two carboxyl groups occurs depending on conditions (nature of substituent at nitrogen atoms, concentration of dithionite, aerobic or anaerobic atmosphere). The dianion of perylenetetracarboxylic acid diimide (3) is formed.

The reverse stage of transition of tetraanions (2) into the initial cubogen (1) can occur due to air oxygen, as indicated by the influence of the atmosphere (Table 2), as well as due to interaction with oxidation products of dithionite (sulfite anions). The dianion (3) transforms into a solid compound (4) on contact with air oxygen.



Scheme 1. Stoichiometric reaction mechanism of the interaction of DBGCC with sodium dithionite in aqueous-alkaline solution (R = Ph; Cl-Ph; 3,5-$(CH_3)_2$Ph; 3-(CH_3)Ph.

Table 2. Composition of reaction products depending on the nature of the cubogen and reaction conditions.

Item n/a	Cubogen	Aerobic environment		Anaerobic environment	
		Low concentration of dithionite	High concentration of dithionite	Low concentration of dithionite	High concentration of dithionite
1	bis(Cl-Ph)Cub	small amounts of tetraanion	large amounts of tetraanion	small amounts of tetraanion	large amounts of tetraanion
2	bis(BZI)Cub	no products	large amounts of tetraanion + cyclisation product	large amounts of tetraanion	large amounts of tetraanion + cyclisation product
3	bis(3,5-$(CH_3)_2$Ph)Cub	no products	large amounts of tetraanion + small amounts of cyclisation product	small amounts of tetraanion	large amounts of tetraanion + small amounts of cyclisation product
4	bis(Ph)Cub	no products	tetraanion + small amounts of cyclisation product	small amounts of tetraanion	large amounts of tetraanion
5	bis(3-(CH_3)Ph)Cub	small amounts of tetraanion	large amounts of tetraanion	small amounts of tetraanion	large amounts of tetraanion

Table 2 summarises the data on the effect of substituents nature, the concentration of dithionite, and the gas medium in which the reaction proceeds on the relative amounts of the intermediate compound (2) and the final product (3) in solution. The amount of anions formed as a result of the reaction was assessed by the optical density of the solutions at the absorption maxima.

According to our analysis, the reaction either does not proceed or tetraanions are formed in small amounts under aerobic conditions at low concentrations of reducing agent. At high concentrations of sodium dithionite, tetraanions are formed in both inert atmospheres and airborne atmospheres. The cyclisation of tetraanions with the formation of the perylene cycle proceeds in appreciable amounts only in the case of bis(3,5-$(CH_3)_2$Ph)Cub and bis(BZI)Cub. This is apparently due to the size of substituents at nitrogen atoms.

Conclusions and recommendations

As a result, the reaction of interaction of N,N´-substituted diimides of binaphthylhexacarboxylic acid (cubogenes) with sodium dithionite in aqueous-alkaline solution, tetraanions of the initial compounds are formed at the first stage of the reaction. Further they can either transfer to the initial state or lose two groups of CO_2 and transform into dianions of N,N'-substituted diimides-3,4,9,10-perylenetetracarboxylic acid.

The results obtained should be taken into account in the technology of finishing textile materials using cubogenes, as well as in liquid-phase preparation of thin films of photoactive materials based on perylene derivatives.

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