The Austrian Journal of Technical and Natural Sciences, No 7–8
ISSN 2310-5607

PREMIER Publishing
ppublishing.org

DOI:10.29013/AJT-25-7.8-25-29

SYNTHESIS AND PROPERTIES OF AZO COMPOUNDS BASED ON SOME AROMATIC AMINES WITH β-NAPHTHOL

Saparbayev Suroj Rustamovich [1], ***Yuldasheva Muhabbat Razzoqberdievna*** [1]

[1] The National University of Uzbekistan named after Mirzo Ulugbek, Tashkent

Cite: *Saparbayev S. R., Yuldasheva M. R. (2025). Synthesis and Properties of Azo Compounds Based on Some Aromatic Amines With B-Naphthol. Austrian Journal of Technical and Natural Sciences 2025, No 7–8.* https://doi.org/10.29013/AJT-25-7.8-25-29

Abstract

In this article, the synthesis of azo compounds with β-naphthol based on aromatic amines with p-substituents was studied. The reaction conditions, physicochemical properties of azo compounds and the structure of the resulting products were determined by spectroscopic methods. Based on the results obtained, the effect of substituents on the product yields was studied. The relevance of the topic is that complexes of the obtained azo compounds with metals (Ni^{2+}, Cu^{2+}) were obtained. The following azo dyes combine with metals to form bidentates.

Keywords: *β-naphthol, p-Br-aniline, p-anisidine, HPLC, IK, AAS*

Introduction

Azo compounds are widely used in the chemical industry, especially in the synthesis of dyes and biologically active compounds. In analytical chemistry, they are used as ligands for the synthesis of metal complexes. Organic coatings that are mainly resistant to metal corrosion are obtained. The synthesis of azo dyes is one of the most interesting reactions in organic chemistry. It not only has useful properties in everyday life, namely as dyes and pigments (Al-Tohamy R., 2022, Alsantali R. I., 2022) but also has a unique mechanism that has long been an important example in undergraduate organic chemistry textbooks (Smith, J.G. 2019). Azo compounds are an important part of dyes, food additives, pigments, indicators, textiles, pharmaceuticals, cosmetics and therapeutics (Al-Rubaie L. A., 2012, Jarad A. J., Majeed I. Y., Hussein A. O., 2018). They have also been used recently as precursors in the synthesis of various products (Zhao R., 2011) and corrosion inhibitors (El-Wakiel N. A., 2016, Raja P. B., 2016).

Azo compounds have a nitrogen and nitrogen double bond as chromophores. These dyes are formed by taking a diazonium salt and attaching it to a strongly activated aromatic ring (Valiulin R., 2020). Azo dyes were synthesized by a solvent-free room temperature grinding method using magnetic Fe3O4 nanoparticles (Zhang Y., 2011). Azo dye polymers were prepared by oxidative polycondensation reaction of naphthol-based azo dyes in aqueous medium using NaOCl oxidant. According to the results, it was found that the durability properties of azo dye monomers are better than those of polymers (Gür M., Kocaokutgen H., 2007). Mono-azo dyes are the most important type

of azo dyes. They are used in various fields: catalysis (El-Ghamry H. A. et al., 2023) supramolecular systems (Ferreira G. R., 2015 p. 13–20, Dinçalp H., 2007, p. 11–24) and metal sensors (Almáši M., Vilkova M., Bednarčík J., 2021) textile and fiber dyeing, which is mainly due to their adsorption capacity and absorption capacity. The remarkable biological activity of azo ligands with O and N donor atoms is known for their excellent chelating ability to all types of metal ions. Azo ligands can be used to form complex compounds containing several metal ions. However, the properties of the complexes produced depend on the type of metal ion used (Zhao R., 2011, p. 3805–3809, Kohl M., 2023, p. 1276) Azo ligand complexes have various applications in medicine, including antitumor (Ferreira G. R., 2015, p. 13–20), antimicrobial (Dinçalp H. et al., 2007, p. 11–24, Abd El-Lateef H. M. et al., 2024, p. 138016) and anti-inflammatory agents (Kasare M. S. et al., 2019, p. 3311–3323).

Experimental part

The M-560 device was used to measure the liquidus temperature of the obtained product. IR spectra IRAffininity-1S, Shimadzu corporation, 2020 Range: 350–7800 cm^{-1}, HPLC: LC-20 Prominence, Shimadzu corporation, Japan, 2020 Range: 190–600 nm, AAS: Atomic absorption spectrophotometer AA-7000, Shimadzu Corporation, Japan, 2020.

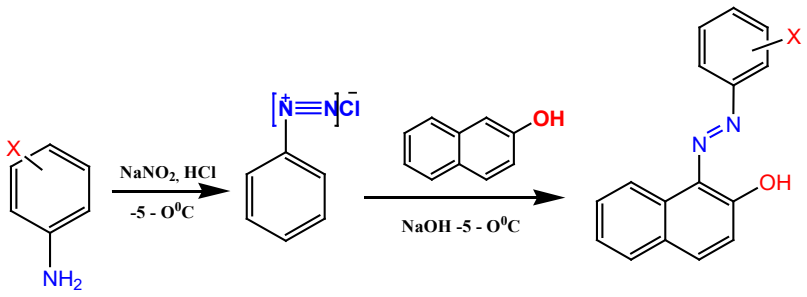
Results and discussions

Synthesis method

During the experiments, aromatic amines (p-anisidine, p-bromoaniline) with various substituents in the p-position were selected. The synthesis of the azo compound was carried out via the azo addition reaction. The azo addition reaction proceeds in the second stage according to the electrophilic mechanism. First, a diazotization reaction occurs, in which a diazonium salt is formed. In the second stage, a weak alkaline solution of β-naphthol with the diazonium salt undergoes an azo addition reaction. These processes are carried out at a temperature of –5–0 °C.

The obtained product was re-filtered with water and recrystallized and purified. The structure of the products was determined using modern physical and chemical methods. Some physicochemical parameters of the azo compounds obtained on the basis of the synthesis. Obtained: (1-(4-methoxyphenylazo-)-2-naphthol) red, Melting point=139–142 °C, Yield 98.6%, Rf=0.65 (1-(4-bromophenylazo-)-2-naphthol) dark red Melting point=170–173 °C, Yield 94.7%, Rf=0.77. TLC (System: EtAc: Pet.ester(1:3)) determined the Rf values. The purity of the obtained azo compounds was checked by HPLC: (1-(4-bromophenylazo-)-2-naphthol) at 8.49 min. we can see peaks with 95.49% purity, (1-(4-methoxyphenylazo-)-2-naphthol) at 14.418 min. we can see peaks with 99.986% purity.Reaksiyaning umumiy sxemasi:

Scheme 1.



X= p-NO_2, p-OH, p-CH_3, p-OCH_3, p-AAB, p-Br, p-COOH, p-aminosalitsil k-ta(PAS)

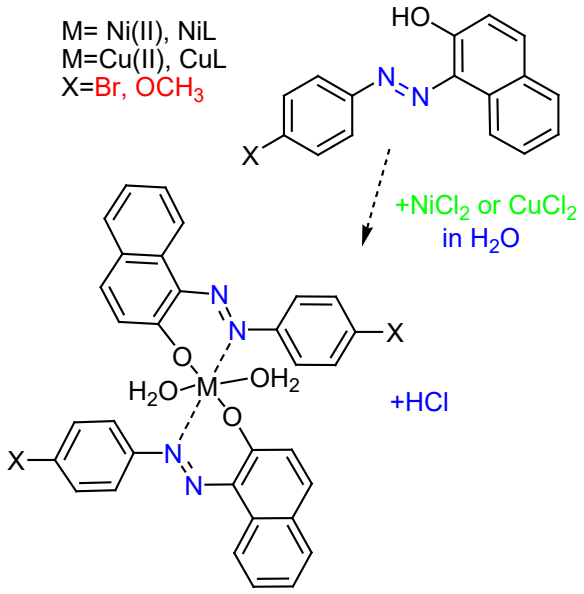
For the synthesis of complexes based on azo compounds, the salt and ligand were dissolved in ethanol in a 1:1 ratio and 1–2 drops of TEA were added to the solution to give an alkaline medium. The solution was heated at boiling temperature for 3 hours

and purified by filtration, washed with water. The IR spectra of the complexes of the obtained azo compounds (1-(4-bromophenylazo-)-2-naphthol), (1-(4-methoxyphenylazo-)-2-naphthol) with Ni(II) and Cu(II) salts are presented:

Table 2.

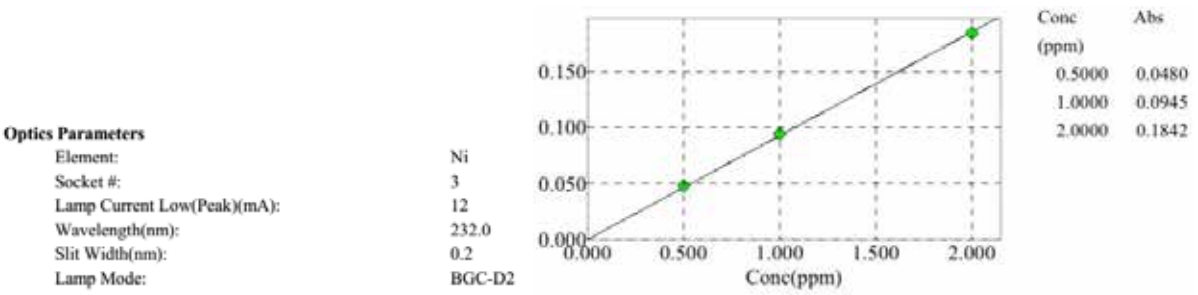
Azocompound	ν(OH)	ν(–N=N-)	ν(M-O)	ν(M-N)
p-BrPAN	3543 sm^{-1}	1496 sm^{-1}	–	–
p-BrPAN-Ni(II)	3057 sm^{-1}	1593 sm^{-1}	663 sm^{-1}	586 sm^{-1}
p-BrPAN-Cu(II)	3444 sm^{-1}	1595 sm^{-1}	603 sm^{-1}	545 sm^{-1}
p-MPAN	3442sm^{-1}	1492 sm^{-1}	–	–
p-MPAN Ni(II)	2837 sm^{-1}	1595 sm^{-1}	620 sm^{-1}	547 sm^{-1}
p-MPAN-Cu(II)	3321 sm^{-1}	1546 sm^{-1}	580 sm^{-1}	559 sm^{-1}

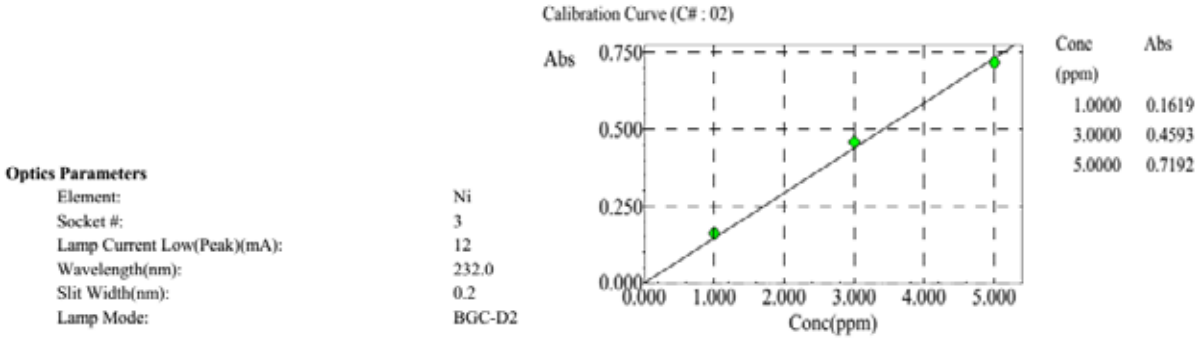
Scheme 2.



The following are the atomic adsorption analysis spectra of the complex formed by (1-(4-bromophenylazo-)-2-naphthol) with Ni(2^{+}) ion. The table shows the atomic adsorption analysis (AAS) of the amount of nickel (Ni^{2+}) ions. The absorptions in the 232 nm region corresponding to Ni(2^{+}) were found to be 14.0% nickel (Ni^{2+}) in the sample. For Cu 2^{+}, the corresponding absorption was determined in the 324.8 nm region and was 16.8% quantitatively. The advantage of this analysis is that it shows the same element (metal) in the composition with high accuracy.

Figure 1.





Conclusion

In this work, the synthesis of azo compounds with β-naphthol based on aromatic amines with substituents in the p-position was carried out. The purity and structure of the obtained products were determined using HPLC and IR spectra. Metal complexes of $Cu2^{+}$ and $Ni2^{+}$ salts of the obtained azo compounds were obtained. Structure of the complexes The composition and amount of metals in the complex were determined using AAS

References

Al-Tohamy R. et al. A critical review of dye wastewater treatment: ecotoxicological and health issues of textile dyes and possible approaches to restore environmental safety // Ecotoxicology and Environmental Safety. 2022. – T. 231. – P. 113160.

Alsantali R. I. et al. Various azo dyes: a comprehensive review of recent advances in biological and industrial applications // Dyes and Pigments. 2022. – T. 199. – 110050 p.

Smith, J. G. Organic Chemistry, 6th ed.; McGraw-Hill Education, 2019.

Al-Rubaie L. A. A. R., Mhessn R. J. Synthesis and characterization of azo dye para red and new derivatives //Journal of Chemistry. 2012. – T. 9. – No. 1. – P. 465–470.

Jarad A. J., Majeed I. Y., Hussein A. O. Synthesis and spectral studies of heterocyclic azo dye complexes with some transition metals // Journal of Physics: Conference Series. – IOP Publishing, 2018. – T. 1003. – No. 1. – 012021 p.

Zhao R. et al. One step synthesis of azo compounds from nitroaromatics and anilines // Tetrahedron letters. 2011. – T. 52. – No. 29. – P. 3805–3809.

Kohl M. et al. New Azo dyes-based Mg complex pigments for optimizing the anti-corrosion efficiency of Zinc-pigmented epoxy ester organic coatings // Coatings. 2023. – T. 13. – No. 7. – 1276 p.

El-Wakiel N. A. Synthesis and characterization of azo sulfaguanidine complexes and their application for corrosion inhibition of silicate glass // Applied Organometallic Chemistry. 2016. – T. 30. – No. 8. – P. 664–673.

Raja P. B. et al. Reviews on corrosion inhibitors: a short view // Chemical Engineering Communications. 2016. – T. 203. – No. 9. – P. 1145–1156.

Valiulin R. Organic Chemistry: 100 Must-Know Mechanisms // Organic Chemistry: 100 Must-Know Mechanisms. – De Gruyter, 2020. – P. 97–98.

Zhang Y. et al. An environmentally friendly approach to the green synthesis of azo dyes with aryltriazenes via ionic liquid promoted CN bonds formation // Dyes and Pigments. 2018. – T. 158. – P. 438–444.

Gür M., Kocaokutgen H., Taş M. Synthesis, spectral, and thermal characterizations of some azo-ester derivatives containing a 4-acryloyloxy group // Dyes and Pigments. 2007. – T. 72. – No. 1. – P. 101–108.

El-Ghamry H. A. et al. A series of nanosized Cu (II) complexes based on sulfonamide azo dye ligands: an insight into complex molecular structures, antimicrobial, antitumor and cata-

lytic performance for oxidative dimerization of 2-aminophenol //Applied Organometallic Chemistry. 2023. – T. 37. – No. 2. – e6978 p.
Ferreira G. R. et al. Supramolecular compounds of azo dyes derived from 1-phenylazo-2-naphthol and their nickel and copper complexes // Supramolecular Chemistry. 2015. – T. 27. – No. 1–2. – P. 13–20.
Dinchalp H. et al. New thiophene-based azo ligands containing azo methine group in the main chain for the determination of copper (II) ions // Dyes and Pigments. 2007. – T. 75. – No. 1. – P. 11–24.
Almáši M., Vilkova M., Bednarčík J. Synthesis, characterization and spectral properties of novel azo-azomethine-tetracarboxylic Schiff base ligand and its Co (II), Ni (II), Cu (II) and Pd (II) complexes // Inorganica Chimica Acta. 2021. – T. 515. – 120064 p.
Abd El-Lateef H. M. et al. Design, synthesis of new mixed azo-hydroxyquinoline complexes; in vitro anti-inflammatory, antifungal, antibacterial, theoretical, and molecular docking interactions Investigation // Journal of Molecular Structure. 2024. – T. 1307. – 138016 p.
Kasare M. S. et al. In-vitro antibacterial activity of Ni (II), Cu (II), and Zn (II) complexes incorporating new azo-azomethine ligand possessing excellent antioxidant, anti-inflammatory activity and protective effect of free radicals against plasmid DNA // Synthetic Communications. 2019. – T. 49. – No. 23. – P. 3311–3323.

submitted 15.08.2025;
accepted for publication 29.08.2025;
published 29.09.2025

Contact: surojsaparbayev70@gmail.com