

 <p>ISSN NO. 2320-5407</p>	<p>Journal Homepage: -www.journalijar.com</p> <h2 style="text-align: center;">INTERNATIONAL JOURNAL OF ADVANCED RESEARCH (IJAR)</h2> <p style="text-align: center;">Article DOI:10.21474/IJAR01/9135 DOI URL: http://dx.doi.org/10.21474/IJAR01/9135</p>	 <p>INTERNATIONAL JOURNAL OF ADVANCED RESEARCH (IJAR) ISSN 2320-5407 Journal Homepage: http://www.journalijar.com Journal DOI:10.21474/IJAR01</p>
---	--	---

RESEARCH ARTICLE

SYNTHESIS AND STUDY OF DYEING PROPERTIES OF HETEROCYCLIC DISPERSE DYES DERIVED FROM 2- AMINO BENZOTHAZOLE.

Hadiza Jibril Abdullahi.

Federal college of education (technical) bichi school of science education department of chemistry p.m.b 3473.

Manuscript Info

Manuscript History

Received: 24 March 2019

Final Accepted: 26 April 2019

Published: May 2019

Key words:-

Azo, disperse dyes,
diazotization, coupling, fastness
properties.

Abstract

Monoazo heterocyclic disperse dyes were synthesized from 2- Amino benzothiazole by diazotization of 2- Aminobenzothiazole and coupling with 1-Naphthylamine, 1- Naphthol and 2-Naphthol. The synthesized dyes were applied to nylon, polyester and acrylic fabrics by Drapkira's method. The fastness properties of the dyed fabrics to light and wash were evaluated, other physical characteristics of the dyes such as melting temperature, and light absorption in the visible spectrum has also been determined. It was observed that these dyes have high wash fastness for the fabrics while their fastness to light is poor and they all absorbed at the visible region of the spectrum.

Copy Right, IJAR, 2019,. All rights reserved.

Introduction:-

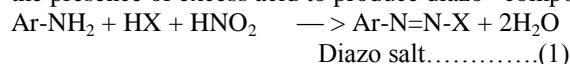
The discovery of disperse dyes was made in 1924 by James Badiley and Holland Ellis but due to their lack of substantivity for natural fibers, their chemistry developed in 1950s, when secondary cellulose acetate and polyester came in to existence. Their use is primarily for synthetic fibers such as nylon, acrylics, polyester, cellulose acetate and also for thermoplastics[1]. Dyes possess color because a. they absorb light in the visible spectrum b. have at least one chromophore (color bearing part) c. have conjugated system d. exhibit resonance of electrons which is a stabilizing force in organic compounds. Dyes also contain auxochromes which are color helpers such as amino and hydroxyl groups. [2] The raw materials employed in the manufacture of synthetic dyes should not involve compounds known to pose health risk which include a large group of aromatic amines.[3] Synthetic dyes are either cancer suspect agents or established mutagens in the standard salmonella mutagenicity assay[4].

Disperse dyes are compounds of low water solubility, non ionic and hydrophobic in nature which falls in to three chemical classes namely Nitrosylamine, Azo and Anthraquinone. Almost all contain hydroxyl, amino or amino substituted groups, are small in size and vaporize when heated. [5] These dyes are generally applied in the form of dispersions in an aqueous medium at a temperature above the boil in an enclosed pressurized dyeing machine or by the use of carrier. Heterocyclic disperse dyes are derived from heterocyclic amine compounds such as 2-aminobenzothiazole termed as diazo compounds which diazotized in to diazonium ion and coupled with coupling component such as aniline, 2-naphthol to produce an Azo compound[6]. The heterocyclic ring may consist of carbon atoms together with either S, O or N. They generally possess good dyeing properties and good fastness properties to wash and light. They had special properties like heterocyclic sulphur compounds which have thermal stability while 5-hydroxy benzimidazoles possessed antioxidant properties. Heterocyclic disperse dyes are applied in the form of dispersions from an aqueous medium at a temperature above the boil in an enclosed pressurized dyeing machine or

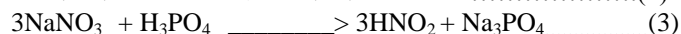
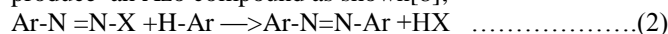
Corresponding Author:-Hadiza Jibril Abdullahi.

Address:-Federal college of education (technical) bichi school of science education department of chemistry p.m.b 3473.

by the use of a carrier [7]. Diazotization reaction; is the reaction of a primary aromatic amine with a nitrous acid in the presence of excess acid to produce diazo compound represented by the equation



Where Ar is a mononuclear or polynuclear aromatic radical; HX is a strong monobasic acid. Diazotization may be performed in aqueous solution, mineral acid, suspensions or in organic solvent, based on the solubility and basicity of the aryl amine to be diazotized. Coupling should immediately follow diazo reaction since the compound are unstable at room temperature and their isolation being not required. The coupling reaction involve coupling the diazonium salt to a compound possessing an active hydrogen atom bond to a carbon atom such as Naphthol to produce an Azo compound as shown[8];



The ease with which coupling reaction occur depend on the nature of the substituent group in the coupling components, for example negative substituent's in the coupling components such as halogen tend to retard coupling while lower alkyl or alkoxy group substituted in ortho or meta position to an amino group may promote coupling. Hydroxyl derivatives are couple under alkaline condition while amine derivative are couple under acidic condition[9]. Dyes in solution absorb in the visible region of the spectrum ranging from 400nm -750nm wavelength, since dyes are colored compounds. Color fastness properties describes the color of dyed or printed textiles to the various agencies to which it may be exposed during processes following coloration and during their subsequent useful life, agencies such as light, washing, bleaching, perspiration etc. Breakdown of the colorant and shade of the colorant inside the fiber, chemical structure of the fiber among other factors affects color fastness [10]. The scale for specifying color fastness to washing and to other agencies excluding light is the 1-5 Grey scale. 9 Fastness to natural day light is a test of the behavior of the colored sample under actual condition of artificial light source with spectral distribution as near as possible to day light. The scale for specifying color fastness to light is 1-8 scale for light fastness [11]. These dyes generally tend to have good to fair fastness properties to wash and light. Although disperse dyes have low water solubility it is accepted that dyeing takes place from a monomolecular dilute solution of the dye in water, the concentration of which is maintained by the continuous dissolution of solid dye from the finely dispersed dye particles in suspension where adsorbed dye diffuses mono molecularly in to the fiber. Dyeing also occurs if the fiber is suspended above a heated dye powder, showing that the transfer via the vapor phase is also possible with disperse dyes[12].

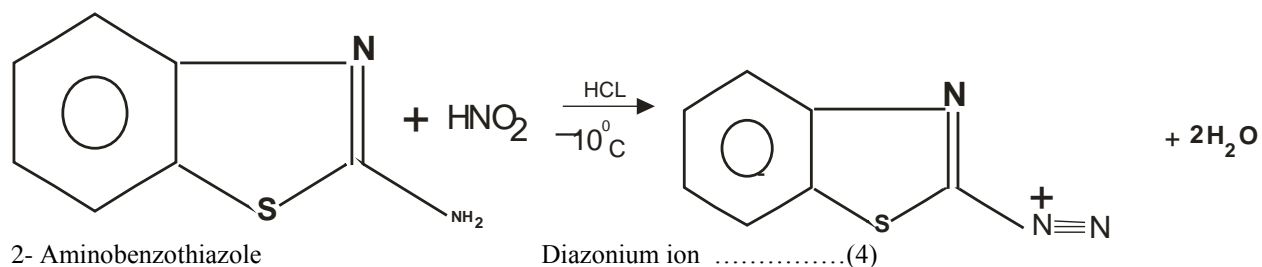
Experimental materials

The chemicals and solvents used in this work were of Analar grade. All the glass wares used were washed thoroughly with distilled water and dried in an oven. Weighing was carried out on electric metler balance, model AB 54.

Reagents used include sodium nitrite(NaNO_2), Sulphanic acid ($\text{NH}_2\text{SO}_3\text{H}$), Phosphoric acid (H_2PO_4), concentrated H_2SO_4 , dilute NaOH, Butan-1-ol, Ethyl acetate, Pyridine, Ethanol, Matexil DA, NN, Sodium dithionite, ECE reference detergent(4g/L NaOH), distilled water. Diazo component, (2-Aminobenzothiazole) and coupling components; 1-Naphthylamine, 2-Naphthol and 1-Naphthol.

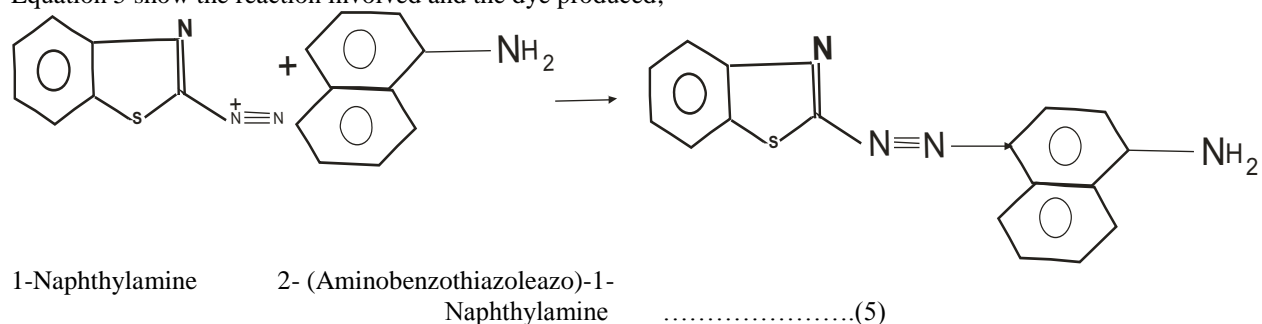
Methods:-

The diazotization of 2-Aminobenzothiazole was carried out by following Drapkira's method. 1.5g 2-Aminobenzothiazole was dissolved in 15ml of phosphoric acid. A solution of 0.69g sodium nitrite in 4ml of water was added to the diazo component while stirring for 1 hour at -10°C . The mixture was maintained at this temperature for further 0.5 hour. Sulphanic acid was added to decompose excess sodium nitrite until no nitrous oxide was produced; starch iodide paper which turns blue in the presence of the nitrous oxide gas was used to test for the presence of the gas. The reaction involved was shown in equation 4[13].

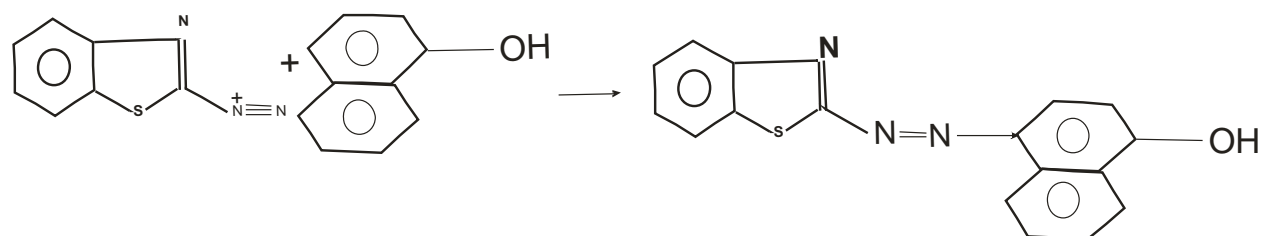


The diazonium ion prepared was used without delay. 1.4319 g of 1-Naphthylamine was added to 3ml of concentrated sulphuric acid in 100ml of water and cooled -10°C . The diazonium salt of 2-Aminobenzothiazole was then added while stirring and the mixture was maintained at that temperature for 4 hours. 200ml of water was then added to the mixture and the dye precipitated out of the solution at PH 3 by the addition of 10 percent aqueous solution of sodium hydroxide.

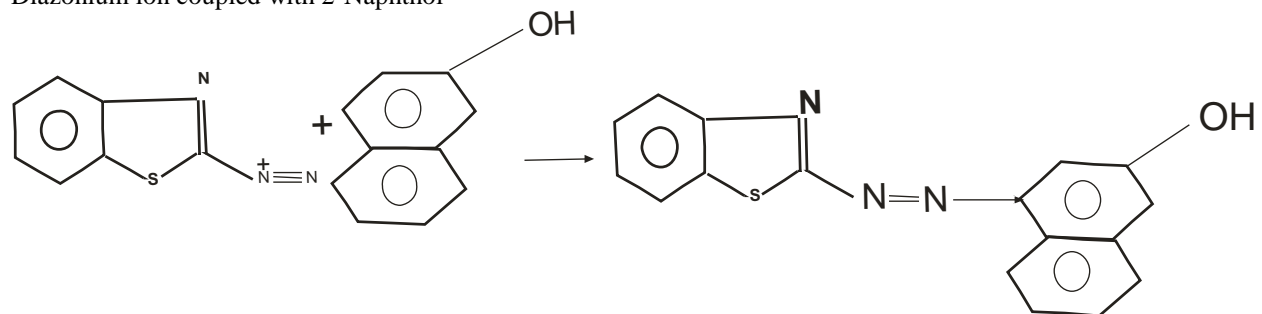
Equation 5 show the reaction involved and the dye produced;



Coupling with 1- and 2-Naphthols; To 1.44g each of 1- Naphthol and 2-Naphthol contained in a beaker, 100ml of 2M Sodium hydroxide solution was added to form a solution. The diazonium salt was gradually added to the mixture while stirring at -10°C and the mixture maintained at this temperature for 2.5 hours. 20ml of water was added to the mixture and the dye precipitated out of the solution. The reactions shown by equations 6 and 7



Diazonium ion coupled with 2-Naphthol



Recovery and purification of the dye; Dyes derived from 1-Naphthylamine and 1- and 2- Naphthol were filtered and air dried. The dyes were purified by re crystallization and their purity ascertained by thin layer chromatography (TLC). Melting points of the purified dyes were determined. Spectroscopic absorption measurements were also carried out.

DYEING METHODS: The dye bath was prepared with 30mg of the dye in 100ml of water. Polyester was dyed in a Good brand –Jeffrey's dyeing machine while nylon and acrylics were dyed in a water bath.

Dyeing of Polyester; 30mg of each dye was dissolved in 5ml Ethanol, 1ml of Matexil DA-NN (a dispersing agent) was added and the dye bath made to 100ml with distilled water. 1g of Polyester yarn was entered in to each dye bath at 60°C. The temperature was then raised to 120°C within 30 minutes and dyeing continued for 80 minutes. The dye bath was then cooled and the fiber rinsed thoroughly with hot water and then with cold water. The dyed substrates were reduction cleared to remove any surface color by treating with Sodium hydroxide (2g/L), Sodium Odithionite (2g/L) and matexil DA-NN (0.1%).

In the dyeing of nylon and acrylics, the dye bath was prepared as for the polyester dyeing and 1g of each fabric were placed in to the dye bath at 40°C. The temperature was then gradually raised to 100°C within 30 minutes. The dye bath was then cooled slowly and the substrate rinsed with cold water. Reduction clearing of the fabrics was done as in that of polyester.

Determination of color fastness to washing;

0.5g each of the dyed substrates were treated in a solution of ECE reference detergent (4g/L NaOH) at 50°C for 30 minutes at a liquor ratio 100:1. A wash wheel from Dinting Textile Engineering was used for the treatment. The color fastness assessment was carried out using the Grey scale for change of color.

Determination of color fastness to light:

Small piece of each dyed substrate was cut and mounted on a piece of cardboard. The blue wool standards [numbered 1-8] were mounted on the card along with the dyed samples. About one third of the samples area was covered by a strip of card. The samples were exposed to the mercury-tungsten lamp in a micro scale light fastness tester. The effective humidity was maintained with distilled water. The samples were exposed for three weeks until the light fastness of each sample could be evaluated.

Results:-

Table1:-Indicate Dye yield, melting point, λ max molecular weight and dye appearance

DYE	MOL.WT	% YIELD	M.P °C	DYE APPEARANCE	λ MAX(nm)	
2-Amino (benzo thiazole azo)- 1-Naphthylamine	305	91.605	180°C	Shiny light brown Crystals	DMF 410	Ethanol 520
2-Amino (benzothiazole azo)-1-Naphthol	306	76.639	70°C		610	580
2-Amino (benzothiazole azo) -2-Naphthol	306	90.746	70°C	Dark brown crystals Red crystals	490	570

Table 2:-Shows colour and colour fastness ratings of the dyes on three fibers (polyester, nylon and acrylic) by the use of 1-5 Gray scale for specifying colour fastness to washing where the highest number on the scale indicates maximum fastness

Dyes	colour and fastness	polyester	Nylon	Acrylic
Rating				
1	Colour	Yellow (L)	Yellow (L)	Yellow (L)
	Fastness	wf= 4	wf=5	wf=4
	Rating	Lf = 3	Lf=2	Lf = 4

2	Colour	Brown (L)	Blue (M)	Blue (L)
	Fastness	wf= 3	wf =3	wf =3
	Rating	Lf = 2	Lf =3	Lf = 3
3	Colour	Red = (H)	Red = (H)	Orange
	Fastness	wf = 4	wf =2	wf =3
	Rating	Lf = 4	Lf =3	Lf = 4

Key

Wf = wash fastness H= Heavy depth of shade Lf =light fastness M= medium depth of shade L= Low depth of shade

= Dye derived from 1-Naphthylamine

= Dye derived from 1-Naphthol

=Dye derived from 2-Naphthol

Discussion:-

From the results shown in table 1, dyes derived from 1-Naphthol and 2- Naphthol have same molecular weight and melting point temperatures of 70⁰c while the dye derived from 1- Naphthylamine has the highest melting point temperature of 180⁰C which could be attributed to the nature of the substituent groups. The λ_{\max} of the dyes varies in the two solvents. Di methyl formamide seems a better solvent for dyes derived from 1-Naphthol and 2- Naphthol than ethanol, while ethanol is a better solvent for the dye derived from 1-Naphthyl amine. The dye derived from 1-Naphthylamine shows high wash fastness for polyester, nylon and acrylic while dyes from 1-Naphthol and 2-Naphthol have well to fair wash fastness for polyester and acrylic but poor wash fastness for nylon. The high wash fastness was due to the state of the colorant inside the fiber which are in form of large particles of water insoluble colorant. Another factor is the chemical structure of the fiber and its physical characteristics. From table one all the dyes have fade due to poor light fastness for all the fabrics after three weeks of exposure where some have been decompose in to colorless and some in to differently colored fabrics. Factors which account for these observations are one or more of the followings breakdown of the colorant inside the fiber in to colorless or differently colored compounds, detachment of the modified colorant from the fiber, chemical structure of the fiber is related to the resistance of the fiber to chemical or photochemical attack, state of the colorant inside the fiber and presence of foreign substances other than the colorants such as after treatment agents etc.

Conclusion And Recommendation:-

Dyes from 1-Naphthol and 2-Naphthol have same melting point temperatures, same molecular weight and formula but different appearance and have different wavelength of absorption all these changes were due to position of the hydroxyl group in each dye molecule. The wavelength of absorption of a dye in a given solvent is influenced by the nature of the solvent since same dye has different wavelength of absorption in different solvents. Nature of substituent group also influenced change in properties since dye from 1-Naphthylamine and those from 1-Naphthol are of different physical appearance and absorb at different wavelength. It was observed that heterocyclic disperse dyes have high wash fastness for polyester, nylon and acrylics fibers while their fastness to light is poor and that all the dyes absorbed at the visible region of the spectrum.

References:-

1. Krik-othmer.[2014].encyclopedia of chemical technology.4ed .new york; john willey and sons pp 673
2. Abrahat en.[1977].dyes and their intermediates. New york chemical publishing company 1-12pp science direct 85
3. Anon. [1996]ecological and toxicological association of dyes and pigments manufacturers. Textile chemists and colorist. Etad information notice no.6 28[4]11.pubmed.
4. Manon dm,ames bn [1983]revised methods journal of american chemical society 10.106pp
5. Venkataraman k.,[2013]the chemistry of synthetic dyes .india ;elsevier 10,241pp
6. Prajapati a.k,bonde n.l,[2016]journal of chemical science 5;118,203pp
7. P.o nkeonye [1989].fundamental principles of textiles dyeing, printing and emishing.amazon.co.uk.
8. Metwally m.a., bondock s., [2015] international journal of modern organic chemistry 1 165pp.
9. R.k fourness[2008].the disperse dyes their development and applications .journal of the society of dyers and colourist.72 issue 11
10. Gregory .p. [1986] azo dyes. Encyclopedia of chemical technology 2nd edition 7 45-56 pp.

11. Mokhtari j., akbarzadeh a., [2009] journal of science and engineering **34** 63pp.
12. Sakohama k.j., bello k.a.,[2012]open journal of applied science **2** 54pp.
13. Morrison, boyd [1987].organic chemistry third edition, publishers allen and bacon inc. Lincoln lin united kingdom.