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Synthesis, Generic Dyeing of Nindigo Derivatives on Unmodified Polypropylene; First Time Application in Dyeing Technology

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Hydrophobic nindigo dyes were designed and successfully synthesized from indigo by reacting with substituted anilines in a simple route. Four nindigo dyes were fruitfully analyzed by ¹H NMR ¹³C NMR, electronic and mass spectrometry. Here, we have firstly introduced these nindigo dyes into dyeing area for the dyeing of unmodified polypropylene fiber in aqueous medium. Dyeing experiments has been performed by using didodecyldimethylammonium bromide dispersing agent to make dye-dispersant complex for enhancing the dispersion of dyes. All the dyes were displayed profound blue color hue on polypropylene. The dyeing efficiency and dye fixation order of the nindigo dyes are $3d > 3c \ge 3b > 3a$. Compound 3d having higher the hydrophobic character ($\log P = 7.04$), so greater the dye ability on the polypropylene fiber and exhibiting deep coloration.

Keywords: Nindigo Dyes, Hydrophobicity, Dyeing, Polypropylene Fiber. Delivered by Ingenta

1. INTRODUCTION

Polypropylene fibers are widely used in industrial and textile applications due to its intrinsic, attractive properties such as inexpensive, resilience, resistant to chemicals and abrasion.¹⁻⁴ The major application aspects are; this fiber does not absorb moisture; it helps quick transport of moisture required applications as babies ever-dry nappies. Thermal conductivity of PP fiber is very low hence, it is used in thermal wear. The density of PP is 0.90-0.92, it helps for the usage in light weight sportswear. Polypropylene possesses hydrophobic character due to its aliphatic and stereo-regular isotactic polymeric structure.⁵ Because of the extreme hydrophobic character of the polypropylene, it is very difficult to dyeing of these fibers at any dyeing systems.⁶ Hence, several investigations have been reported to enhancing the dye ability on isotactic polypropylene. The methods included (a) spin coloration with organic and inorganic pigments⁷ however, this method has some limit to the inventor for the developing of various satisfied fashion demands of the current market. (b) Physical modification of polypropylene involves the addition of polar

additives (basic nitrogen atoms) to preferable dyeing with acid dyes,^{8,9} (c) chemical modification involves chlorination/bromination of fiber with chlorine/bromine to react with cationic dyes.^{10, 11} While these two routes are expensive and also mainly effect on the mechanical properties of polypropylene fiber. (d) Dyeing of unmodified fiber method is possible at the established process by selecting super hydrophobic dyes.^{12–15} This process has significant importance over other, since fiber doesn't loss its physical character and having more demand in rising market for its relatively inexpensive fiber.¹⁶

Indigo is well-known blue colored natural vat dye. It is valuable and having unique properties.¹⁷ Furthermore, it has very low chronic and acute toxicity.¹⁸ However, potential applications are limited due to its low solubility. In its leuco form indigo has more dyeing affinity with cotton silk and wool, but for synthetic fibers like polyester and polypropylene indigo has less dyeing affinity.^{19, 20} Reintroduction of natural dyes is considerable interest in textile industry due to increased attention to environmental aspects and sustainability of raw materials and products.^{21, 22} In order to improve the hydrophobic character of indigo dye will challenge us to

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designing of new molecules. Our main aim is to design of novel molecules (nindigo dyes) which imparted super hydrophobic character and have strong dyeing affinity with the hydrophobic polypropylene fiber. Here we have successfully synthesized four super hydrophobic nindigo dyes and were efficiently applied on unmodified polypropylene fibers using conventional dyeing.

2. EXPERIMENTAL DETAILS

2.1. Materials

All the chemicals and commercial reagents were bought from Sigma Aldrich and Alfa Aesar. Solvents were distilled and dried under nitrogen atmosphere before going to use. ¹H NMR and ¹³C NMR spectra were recorded at RT using an AVANCE III spectrometer operating on either 600 MHz and 150 MHz respectively with CDCl₃ as solvent. The chemical shifts (δ) values were given in ppm downfield from an internal standard tetramethyl silane. UV-visible absorption and reflectance spectra were recorded using a Shimadz UV 26000 spectrophotometer. Mass spectra were recorded on a Q-TOF II instrument using electrospray ionization source in the positive mode. Dyeing experiments has been performed by using ACE-6000 T model Infrared dyeing machine.

2.2. Synthesis

Our key task of the present exertion is to develop commercially most acceptable super hydrophobic dyes for polypropylene fabric. For that instance, we have synthesized nindigo dyes and are applied on polypropylene fabric.

Nindigo derivatives synthesis is involved the reaction of indigo **1** with excess of substituted anilines **2(a–d)**, titanium tetrachloride (TiCl₄) and 1,4diazabicyclo[2.2.2]octane (DABCO) in bromobenzene²³ (Scheme 1). Here bromobenzene is required for solubilize indigo. By using this method, we have successfully synthesized four nindigo dyes and corresponding structures were represented in Figure 1.

2.3. General Experimental Procedure for the Synthesis of Nindigo Derivatives 3(a-d)

In a two neck round bottom flask (100 mL) charged with bromobenzene (10 mL), DABCO (2.52 g, 22.896 mmol)





and substituted anilines 2(a-d) (9.523 mmol). To this stirred mixture, solution of TiCl₄ (1 M in toluene, 5.724 mmol, 5.5 mL) was slowly added. After completion of the TiCl₄ addition, solid indigo (1 g, 3.816 mmol) was added to the above stirred mixture. Then reaction mixture was heated to 160 °C for 24 h. After reaction completion [checked by TLC (thin layer chromatography), the Rf value of nindigo is 0.6 in 10% ethyl acetate: hexane]. The hot solution of reaction was directly filtered through celite pad, and several times washed with dichloromethane until the filtrate was colorless. The filtrate was concentrated under reduced pressure. The crude reaction mixture was absorbed with silica and performed a column purification using 3% *n*-hexane and ethyl acetate as eluent to afford dark blue powders **3(a-d)**.

2.3.1. N,N'-([2,2'-Biindolinylidene]-3,3'-Diylidene) Bis(4-Methylaniline) (3a)

Dark Blue solid; Yield: 75% (1.25 g), mp: 290–292 °C; ¹H NMR (CDCl₃, 600 MHz): δ 9.86 (brs, 2H), 7.20–7.14 (*m*, 8H), 7.05–7.01 (*m*, 4H), 6.94–6.90 (*m*, 2H), 6.70– 6.66 (*m*, 2H), 2.34 (*s*, 6H); ¹³C NMR (CDCl₃, 150 MHz): δ 143.3, 132.8, 129.5, 128.7, 123.8, 119.6, 118.9, 117.7, 114.1, 19.9. MS *m*/*z*: 441 [M+H]⁺.

2.3.2. N,N'-[2,2'-Biindolinylidene]-3,3'-Diylidene) Bis(4-Ethylaniline) (3b)

Dark Blue solid; Yield: 73% (1.3 g), mp: 286–288 °C; ¹H NMR (CDCl₃, 600 MHz): δ 9.87 (brs, 2H), 7.21–7.16 (*m*, 6H), 7.15–7.13 (*m*, 2H), 7.08–7.04 (*m*, 4H), 6.95–6.92 (*m*, 2H), 6.71–6.67 (*m*, 2H), 2.64 (*q*, *J* = 7.5 Hz, 4H), 1.23 (*t*, *J* = 7.5 Hz, 6H); ¹³C NMR (CDCl₃, 150 MHz): δ 143.5, 139.3, 129.4, 127.6, 123.8, 119.6, 118.9, 117.7, 114.1, 27.4, 14.7. MS *m/z*: 469 [M+H]⁺.

2.3.3. N,N'-Bis(3-Ethylphenyl)-[2,2'-Biindolinylidene]-3,3'-Diimine (3c)

Dark Blue solid; Yield: 72% (1.28 g), mp: 280–282 °C; ¹H NMR (CDCl₃, 600 MHz): δ 9.88 (brs, 2H), 7.26 (*t*, *J* = 8.0 Hz, 2H), 7.21–7.15 (*m*, 4H), 6.99–6.89 (*m*, 8H), 6.69 (*td*, *J* = 8.0, 1.1 Hz, 2H), 2.62 (*q*, *J* = 7.5 Hz, 4H), 1.20 (*t*, *J* = 7.5 Hz, 6H); ¹³C NMR (CDCl₃, 150 MHz): δ 146.0, 144.6, 129.5, 128.1, 123.9, 122.6, 119.7, 118.1, 117.8, 115.9, 114.1, 27.9, 14.6. MS *m*/*z*: 469 [M+H]⁺.

2.3.4. N,N'-Bis(4-Propylphenyl)-[2,2'-Biindolinylidene]-3,3'-Diimine (3d)

Dark Blue solid; Yield: 66% (1.24 g), mp: 262–264 °C; ¹H NMR (CDCl₃, 600 MHz): δ 9.86 (brs, 2H), 7.20– 7.12 (*m*, 8H), 7.05 (*d*, *J* = 8.0 Hz, 4H), 6.91 (*d*, *J* = 7.9 Hz, 2H), 6.68 (*t*, *J* = 7.7 Hz, 2H), 2.57 (*t*, *J* = 7.5 Hz, 4H), 1.67–1.60 (*m*, 4H), 0.92 (*t*, *J* = 7.3 Hz, 6H); ¹³C NMR (CDCl₃, 150 MHz): δ 143.5, 137.7, 129.5, 128.2, 123.8, 119.6, 118.8, 117.7, 114.1, 36.5, 23.7, 12.7. MS *m*/*z*: 497 [M+H]⁺.

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Figure 1. Structures of nindigo 3(a-d).

2.4. Dve Dispersions Preparation

First to prepare uniform dyes and dispersing agent mixtures, 5% owf amount of synthesized nindigo dyes were separately dissolved in 50 ml of tetrahydrofuran and mixed thoroughly with the solution of DDAB [2 molar ratio compared to the dye amount] in 50 ml tetrahydrofuran (THF).²⁵ The solution mixture was concentrated with the help of rotatory evaporator and dried for 12 h for complete removing of tetrahydrofuran. The mixture was dissolved in water and sonicate for 1 h. Finally, this solution can be used for dyeing.

2.5. Dyeing

The unmodified polypropylene fiber samples (500 mg) were used for dyeing with appropriate 1%, 3% and 5% owf dye dispersion solution at the liquor ratio of 1:50. DDABan used for dye dispersion solution [1, 2 and 3 molar ratio] $^{\circ}$ (130 $^{\circ}$ C) and constant molar ratio of dispersing agent compared to the dve amount. The dve bath temperatures were used in this study is 70 °C, 100 °C and 130 °C. The dye bath temperatures were used in this study is 70 °C, 100 °C and 130 °C. These temperatures were maintained for 1 h at a rate of 2 °C/min. After dyeing the dyeing bath was cooled and then polypropylene fabrics were removed. The dyed fabrics were treated with sodium hydroxide and sodium hydrosulfite (1;1, 2 g/l) at 70 °C for 15 min to completing reducing step.

3. RESULTS AND DISCUSSION

In our experiments we have studied the effect of dyeing temperature, dye concentration and dispersing agent quantity on color strength values.

3.1. Dyeing Temperature Effect on Color Strength (K/S)

Dyeing temperature plays an essential role on color strength values. We have performed the dyeing experiments using three variable (70 °C, 100 °C and 130 °C) temperatures. Here we didn't try the higher temperatures after 130 °C because of the poor thermal property of poly propylene.14 At 70 °C, 100 °C and 130 °C the color strength values of dyed fabrics 7.46, 8.13, 9.38 for compound **3a**, 7.53, 8.84, 9.96 for **3b**, 7.79, 8.93, 10.19

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for 3c, 11.28, 12.56, 13.83 for 3d and 3.01, 3.69, 4.93 for compound 1 respectively (Fig. 2). Based on the results, higher temperature could increase the color strength values, because the dye particles were moved very fast, which enhances the rate of interaction with solvent particles. Therefore, the dyes solubility is increases and more absorption of dye particles on the surface of polypropylene fibers. Meanwhile, it also could enhance freedom of the polypropylene macromolecular chains.³ Finally, 130 °C is the considerable optimized dyeing temperature.

3.2. Dye and Dispersing Agent Concentration Effect on Color Strength (K/S)

To study, dye concentration effect on color strength values, we have performed the experiments using three variable concentrations (1%, 3%) and 5%) at constant temperature (1 molar ratio). Based on the results, we observed that the dye concentration is increased, the color strength values are also increases (Fig. 3). This is due to that on increasing concentration of the dye, more and more dye molecules are available to interact with fiber in the dye bath. Therefore, the rate of absorption is increased as well as color strength values are also increased.



Figure 2. Effect of dye temperature on color strength (K/S) values.



Figure 3. Effect of dye concentration (% owf) on color strength (K/S) values.

On the other hand, we have also studied that the effect of the various amounts of didodecyldimethylammonium bromide (DDAB) dispersing agent (1, 2 and 3 molar ratios) on the color strength values and the corresponding results were showed in Figure 4. At 1 molar ratio of dispersing agent the color strength values are minimum. This is due that in presence of small quantity of dispersing agent, the probability of aggregation of dye particle is very less and also very difficult to formation of stable dispersion. At 2 molar ratio of dispersing agent we have observed maximum color strength values. In contrast, at 3 molar ratio of dispersing agent the color strength values are also slightly decreased, because at this concentration dispersing agent forms highly stable dye dispersion in dyeing liquor to be adsorbed and penetrated into polypropylene fiber from the liquor.¹³



Figure 4. Effect of dispersing agent concentration on color strength (K/S) values.

3.3. Dye Ability

The dye ability of nindigo dyes on unmodified polypropylene fiber mainly depending upon the partition coefficient $(\log P)$. It represents the hydrophobic character of organic compound. By using Chem3D we have obtained $\log P$ values.²⁴ In the dyeing experiments, it is very essential to form stable dispersion of dyes, for that didodecyldimethylammonium bromide surfactant was used. In common organic solvents, this surfactant having similar solubility to the nindigo dyes. Therefore, dye-dispersion complex has been formed from dye and dispersing agent uniform mixture solution. By adding the water and performed ultra-sonication generated vesicle by dispersing of nindigo dyes. Then, the nindigo dye molecules were transferred from surfactant micelle to the surface of the fiber.²⁵ The adsorbed nindigo dyes diffused in to the fiber (Fig. 5). The dve ability is very high for compound 3d when compare to the others 3c, 3b, 3a and compound 1. The log P values for the dyes are 5.37 for 3a, 6.21 for 3b and 3c, 7.04 for 3d and 0.16 for compound 1. Due to highest log P of compound 3d, the distribution rate is high between polypropylene fiber and solution. Therefore, the dye ability is also very high.

3.4. Color Strength (K/S) and Dye Fixation (%)

The color strength (K/S) values of dyed polypropylene fabrics were calculated from the Kubelka-Munk equation by substituting of the reflectance (R%), which is recorded by using Shimadz UV 26000 spectrophotometer.

$$K/S = \frac{(1-R)^2}{2R}$$

Where K is absorbance and S is the scattering.

To calculate the dye fixation (F) percentage values, we have substituted the values of $(K/S)_{\text{extracted}}$ (after extraction) and $(K/S)_{\text{dyed}}$ (after dyeing) in below equation. To measure the $(K/S)_{\text{extracted}}$, first the dyed polypropylene fibers were soaked in acetone (250 cm³) in a beaker and soaked materials were heated at 60 °C for 10 min and temperature gradually raised to boiling temperature (80 °C) and maintained at the boiling temperature for 1 h to yield a dye extracts. The dye extract was left to stand for 30 min at ambient temperature and filtered. The fabrics were removed and dried to measure the color strength values.

$$F = \frac{(K/S)_{\text{extracted}}}{(K/S)_{\text{dyed}}} \times 100\%$$

Dye **3d** had comparative $[(K/S)_{dyed} \ 13.82]$ and $[(K/S)_{extracted} \ 10.93]$ values are significantly higher than the **3c**, **3b**, **3a** and indigo. There is no much differentiation in $(K/S)_{dyed}$ and $(K/S)_{extracted}$ values of **3b** $[(K/S)_{dyed} \ 10.19$ and $(K/S)_{extracted} \ 7.33]$ and **3c** $[(K/S)_{dyed} \ 9.96$ and $[(K/S)_{extracted} \ 7.07]$. The justification for this result, these two dyes are possessing equal hydrophobic character. The percentage of dye fixation for the dyes ranked in

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Figure 5. Dyeing mechanism with nindigo dyes on polypropylene.

the following order $3d > 3c \ge 3b > 3a > 1$ (Fig. 6). This could be attribute to the molecular structure and hydrophobic character of the dyes. If the dye molecules are having more hydrophobic character, the substantivity is increased with the polypropylene fiber, thus the adsorption of dye molecules on fiber is also increased. Therefore, the dye fixation *F* values are also increased. Figure 7 indicated that the before and after dyeing of polypropylene fabrics with our synthesized dyes (3a, 3b, 3c, 3d) and indigo.

3.5. Color Assessment

The color assessments of the dyed polypropylene fabrics were studied using data color 110 spectrometer. The CIELAB coordinates were measured and were represented as L^* (lightness), ΔL^* (difference in lightness and darkness), positive values (+ve) of ΔL^* indicates lighter and negative values (-ve) of ΔL^* indicates darker. C^* (chroma), ΔC^* (difference in chroma) positive values (+ve) of ΔC^* indicates brighter and negative values (-ve) of ΔC^* indicates darker. *h* (hue angle from 0° to 360°), ΔH^* (difference in hue) where positive value (+ve) of ΔH^* indicates yellowish and negative values (-ve)



Figure 6. $(K/S)_{dyed}$, $(K/S)_{extracted}$, and % of *F* values of dyed polypropylene fabrics.

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of ΔH^* indicates reddish. ΔE^* is the total color difference. Color-opponent dimensions a^* and b^* . Positive values (+ve) of the a^* indicates the degree of redness and negative values (+ve) of the a^* indicates degree of greenness, similarly positive values (+ve) of the b^* indicates the degree of yellowness and negative values (-ve) of the b^* indicates degree of blueness. The color strength values were represented with K/S.

The color coordinates were listed in Table I. Based on the results our synthesized nindigo dyes are having good dyeing affinity with unmodified polypropylene fabric with the following statements.

In our study the color lightness L^* values of nindigo dyes varied from 61.38 to 69.52. The compound 3a exhibited lighter and duller than the other compounds. According to the negative values of a^* , the color hues of all the synthesized dyes 3(a-d) along with compound 1 were shifted to greenish direction on the red-green axis. Due to exhibiting the positive values of b^* dye **3d** color hue shifted to yellowish direction on the yellow-blue axis and remaining all the dyes 3a, 3b, 3c and indigo are having negative values of b^* , therefore the color hues were shifted to bluish direction on the yellow-blue axis. According to C^* dye **3b** is brighter than the other compounds. The color strength K/S values of 3d is higher than the 3c, 3b, 3a and 1, because the hydrophobic character of 3d is high. Whenever increasing the length of the alkyl chain $\log P$ value (hydrophobic character) is increased, the dye affinity is also increased with polypropylene. Therefore, the K/S value is increased. For the dyes 3c and 3d the ΔH value is negative that indicates reddish shift whereas the dyes 3a and **3b** the ΔH value is positive that indicates yellowish shift compared to the standard values. Furthermore, ΔL value is negative only for dye 3b which indicates a darker color than standard.

3.6. Color Characteristics

Indigo having poor solubility in most of the organic solvents, but our synthesized nindigo derivatives **3a**, **3b**, **3c**

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Figure 7. Photographs of before and after dyeing of polypropylene fabrics.

and 3d were exhibiting excellent solubility in common organic solvents like benzene, toluene, dichloromethane, chloroform, tetrahydrofuran and acetone. All the nindigo derivatives were exhibited bluish green colored solutions with these solvents. Similar to indigo, nindigo derivatives were also comes under donor-acceptor chromogen category, where the amino groups and imine groups were act as the electron donors and electron acceptors respectively. We have recorded the electronic spectra in tetrahydrofuran solution in the range of 400–800 nm using 1.0×10^{-4} M concentration (Fig. 8). At this concentration the electronic spectra contain maximum absorption band (λ_{max}) at 597 nm, which is results from $\pi - \pi^*$ transitions similar to indigo. In addition, we have observed absorption bands at 667 nm and 732 nm for nindigo dyes 3(a-d), these are mainly results from solution aggregation effects. Here we did not observe any effect on molar extinction

Table I. Color of dyed polypropylene fabrics.

	Color coordinates						CIELAB difference			
Dye	L^*	<i>a</i> *	b^*	C^*	h	K/S	ΔL	ΔC	ΔH	ΔE
3a	61.38	-3.68	-4.81	6.06	232.58	9.38	0.00	0.01	0.02	0.02
3b	61.68	-7.88	-1.99	8.13	194.17	9.96	-0.01	0.01	0.01	0.01
3c	63.20	-4.62	-4.17	6.23	222.07	10.19	0.00	0.00	-0.03	0.03
3d	69.52	-5.06	0.55	5.09	173.78	13.83	0.00	0.02	-0.02	0.03
1	63.23	-1.69	-6.67	6.88	255.75	4.93	0.02	0.02	0.00	0.03



Figure 8. Electronic spectra of nindigo 3(a–d) and indigo.



Figure 9. Reflectance spectra of dyed polypropylene fabrics with Snindigo **3(a-d)** and indigo.

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coefficient and maximum absorption wavelength by changing the position and length (methyl, ethyl, and propyl) of the alkyl groups on the dyes. This is concluded that the optical properties of the dyes are almost similar.

We have also recorded the reflectance spectra of dyed polypropylene fabrics by adopting white standard $BaSO_4$. The reflectance curve minimum value centered around 601 nm for **3a**, 608 for **3b**, 615 nm for **3c**, 603 nm for **3d**, and 658 nm for compound **1** (indigo) (Fig. 9).

4. CONCLUSION

In summary we have successfully designed and synthesized four super hydrophobic nindigo derivatives. These nindigo derivatives were applied dyeing on unmodified polypropylene fiber. Here, we have studied the effect of main operating conditions like temperature, dye concentration and dispersing agent concentrations on color strength values. The optimized dyeing temperature is 130 °C and dispersing agent concentration is 2 molar ratio. In these experiments compound **3d** exhibits excellent color strength and good dye fixation. Because this compound having higher log P (hydrophobicity) value. As increasing the hydrophobic character ability to interact with polypropylene fiber is very high. As far as we know, this is the first report on dyeing of unmodified polypropylene fiber with

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hydrophobic nindigo dyes. These results will be prompted us to further design of new hydrophobic dyes suitable for unmodified polypropylene.

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