

# Dyeing Properties of the Disperse Dyes Containing Cyano Group Based on Benzisothiazole for Polyester Fabrics under Alkali Condition

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**Abstract:** One step dyeing of polyethylene terephthalate (PET) fabrics combining pretreatment and dyeing under the alkali condition was developed for cleaner production. One step dyeing of PET fabrics required that the dye used has good acid and alkali stability. In this paper, dyeing properties of three azo disperse dyes containing cyano group based on benzisothiazole, 3-(4-N-ethoxyl-N-cyanoethyl-phenyldiazenyl)-5-nitro-2,1-benzisothiazole (D1), 3-(4-N-ethyl-N-cyanoethyl-phenyldiazenyl)-5-nitro-2,1-benzisothiazole (D2), and 3-(N-benzyl-N-cyanoethyl-phenyldiazenyl)-5-nitro-2,1-benzisothiazole (D3), were investigated under alkali condition. The results showed that polyester fabrics could be well dyed with D1, D2 and D3 under the acid condition. However, D1 was decomposed while dyeing at the alkali solution. D2 and D3 had excellent color yields under the alkali condition. The acid-alkali stability and the structure change were analyzed by UV-vis spectrum and high pressure liquid chromatography (HPLC). Gaussian 09 program package was used to optimize geometry by B3LYP method and 6-31G (*d*) basis set. The solvation energy of D1 in water was higher than those of D2 and D3. The electron withdrawn effect of the hydroxyl affected the energy gap of HOMO and LUMO orbits. D2 and D3 showed excellent stability in the strong alkali medium. And the dyed polyester fabrics with D2 and D3 at the alkali condition also had good fastness properties.

**Keywords:** PET, Acid-alkali stability, Disperse dyes, Solvation energy, Benzisothiazole

## Introduction

The conventional dyeing of textile fabric is generally performed in water solution, which results in a high consumption of water and energy [1-3]. In the dyeing of PET fabric with disperse dyes, processing of fabric pretreatment (desizing) and aftertreatment (reduction-clearing) are often under alkali condition. However, dyeing process is usually dyed in weak acidic medium. The processing consumes a large amount of chemicals, water, and energy, because of neutral reaction and scouring [4-6]. So, the new dyeing method, one step dyeing of PET fabrics combining pretreatment and dyeing under the alkali condition, was developed. One step dyeing of PET using the alkali stable disperse dyes is a significant cleaner method for combined wet processing [7,8].

The heterocyclic azo dyes have attracted considerable attention due to their versatile applications in textile industry, and many high-technological frontier fields [9-11]. Some heterocyclic dyes containing cyano group not only show excellent photo-chemical properties, but also possess especial stability for acid and alkali and excellent light fastness [12-14]. The acid-alkali stable property of azo dyes has an important significance for developing novel cleaner dyeing method.

In the family of disperse dyes, most disperse dyes for PET fabrics dyeing are not stable under the alkali condition [7,14,15]. The related investigation of the structure and the

acid-alkali stable property of the disperse dyes are scarce. It is well-known that the minimal change of the substituent groups of a given backbone will influence the structure of the azo dye molecules. It could change the dye dipole moment, stability, molecular arrangements, hydration energy and so on [16-19]. The dye molecule stability is closely related with the intermolecular and intramolecular interaction of the dyes. The heterocyclic dyes containing cyano group possess different acid-alkali stability because of the adjacent substituted tail groups.

In this paper, in order to investigate the chemical structure of heterocyclic disperse dyes on the acid-alkali stability, three disperse dyes containing the cyano group based on benzisothiazole, 3-(4-N-ethoxyl-N-cyanoethyl-phenyldiazenyl)-5-nitro-2,1-benzisothiazole (D1), 3-(4-N-ethyl-N-cyanoethyl-phenyldiazenyl)-5-nitro-2,1-benzisothiazole (D2), 3-(N-benzyl-N-cyanoethyl-phenyldiazenyl)-5-nitro-2,1-benzisothiazole (D3), were synthesized. Their dyeing properties under the acid and alkali conditions were discussed, respectively. The energy gaps of LUMO and HOMO of them were analyzed. The intermolecular and intramolecular interactions of them were also discussed.

## Experimental

### Materials

3-amino-5-nitro-2,1-benzisothiazole, N-ethoxyl-N-cyanoethyl-aniline, N-ethyl-N-cyanoethyl-aniline, N-benzyl-N-cyanoethyl-aniline were obtained from Zhejiang Wanfeng Chemical Company, Shaoxing, China. The desized and bleached PET

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fabric was obtained from Shaoxing Jinqiu Textile Company, Shaoxing, China. Other chemicals used were obtained from Shanghai Chemical Reagent Plant, Shanghai, China.

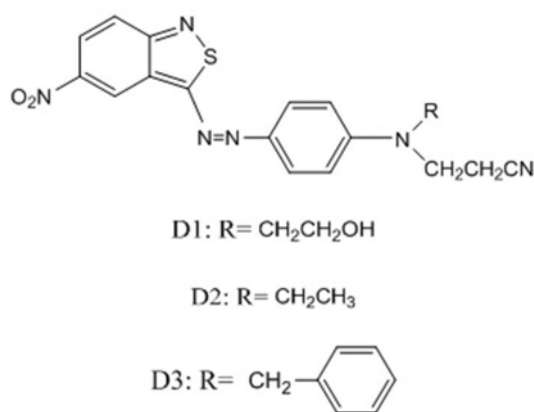
### Synthesis of the Designed Dyes Containing Cyano Group

The chemical structures of the heterocyclic azo disperse dyes containing cyano group based on benzisothiazole are shown in Scheme 1.

The diazotization and the coupling reaction were carried out as described in the references [14,16,18]. 3-amino-5-nitro-2,1-benzisothiazole was diazotized with nitrosylsulphuric acid according to the usual diazo procedure. N-ethoxyl-N-cyanoethyl-aniline, N-ethyl-N-cyanoethyl-aniline, and N-benzyl-N-cyanoethyl-aniline were used as coupling components, respectively. The synthesized dyes were isolated, then filtered and dried in a vacuum oven at 75 °C. The crude dyes were obtained. They were recrystallized from ethanol and were named D1, D2, D3, respectively. FTIR spectrum was measured by NEXUS-670 FTIR Spectrometer (Nicolet Analytical Instruments, Madison WI). <sup>1</sup>H NMR spectrum was recorded on a Bruker AV 400 (Bruker Co., Faellanden, Switzerland). Element analysis for C, H and N were performed on a Vario EL III (Elementar Co., Germany). The melting points of the dyes were obtained with a Koffler hot stage apparatus and were given uncorrected.

D1: The yield was 90.7 %,  $\lambda_{\max}$  580 nm (acetone), mp 147-149 °C. Anal. calcd: C 54.54, H 4.04, N 21.21; found: C 54.34, H 4.10, N 20.98. <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>,  $\delta$ , ppm): 3.26 (s, 1H, OH), 3.99 (m, 4H, -CH<sub>2</sub>-), 4.5 (m, 4H, m, -CH<sub>2</sub>-N- and -CH<sub>2</sub>-O-), 7.36-7.89 (m, 2H, =CH-), 7.92-8.0 (m, 2H, -N=CH-), 8.39 (s, 1H, H of nitrobenzothiazole cycle), 8.88 (s, 1H, H of nitrobenzothiazole cycle), 9.16 (s, 1H, H of nitrobenzothiazole cycle). IR (cm<sup>-1</sup>): 3430, 2923, 2862, 2246, 1597, 1504, 1343, 1142, 1126, 1074, 820.

D2: The yield was 89.4 %,  $\lambda_{\max}$  580 nm (acetone), mp 162-164 °C. Anal. calcd: C 56.84, H 4.21, N 22.21; found: C 55.99, H 4.12, N 21.85. <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>,



**Scheme 1.** Chemical structures of the designed disperse dyes containing cyano group.

$\delta$ , ppm): 1.27 (m, 3H, CH<sub>3</sub>), 2.77 (m, 2H, -CH<sub>2</sub>-), 3.73 (m, 2H, -CH<sub>2</sub>-), 3.90 (m, 2H, -CH<sub>2</sub>-N-), 6.85-6.89 (m, 2H, =CH-), 7.83-7.92 (m, 2H, -N=CH-), 8.30 (s, 1H, H of nitrobenzothiazole cycle), 8.77 (s, 1H, H of nitrobenzothiazole cycle), 9.26 (s, 1H, H of nitrobenzothiazole cycle). IR (cm<sup>-1</sup>): 2923, 2863, 2247, 1598, 1518, 1505, 1343, 1144, 1125, 1074, 823.

D3: The yield was 87.6 %,  $\lambda_{\max}$  567 nm (acetone), mp 178-179 °C. Anal. calcd: C 63.02, H 4.60, N 18.38; found: C 63.07, H 4.52, N 18.27. <sup>1</sup>H NMR (400 MHz, DMSO-d<sub>6</sub>,  $\delta$ , ppm): 2.79 (m, 2H, -CH<sub>2</sub>-), 3.82 (m, 2H, -CH<sub>2</sub>-), 4.00 (m, 2H, -CH<sub>2</sub>-N-), 6.91-6.94 (m, 4H, =CH-), 7.25-8.05 (m, 5H, -N=CH-), 8.27 (s, 1H, H of nitrobenzothiazole cycle), 8.90 (s, 1H, H of nitrobenzothiazole cycle), 9.25 (s, 1H, H of nitrobenzothiazole cycle). IR (cm<sup>-1</sup>): 2923, 2247, 1597, 1515, 1403, 1344, 1328, 1161, 1142, 1124, 1074, 813.

### Dispersion of the Disperse Dyes

The crude dye (75 g), dispersing agent, sodium salt of polycondensated naphthalenesulphonic acid (Dispersant MF), 45 g, and water, 180 ml, were added to a grinding mill (8.5 cm inner diameter), and the mixture was stirred for 15 min. Then grinding material, silica sand (density 2.66 g/cm<sup>3</sup>, fineness 100-150 mesh), 600 g, was added and subsequently grinded for 30 min at 30 °C with 1260 r/min.

### Dyeing of PET Fabric

The dyeing of PET fabric was carried out with the similar way under the alkaline system as described in the reference [7]. The dyeing solution was prepared by using dye, 2 % (o.w.f.), based on weight of fabric. The pretreated fabric was dyed in a PYROTEC-2000 IR dyeing machine, the liquor ratio being 1:10, auxiliary AS-1 1 g/l as leveling agent, and NaOH, 5 g/l. The PET fabric was immersed in the dye bath at room temperature and the temperature was increased to 130 °C at a rate of 1 °C/min. The dyeing was carried out at this temperature for 60 min. After dyeing, the dyed samples were treated for 15 min in sodium hydrosulfite, 0.5 g/l, and sodium carbonate, 1.0 g/l, at 75 °C. Then, all the samples were rinsed with water until the rinsing water was clear and dried.

### Color Measurement

The color yield ( $K/S$ ) and colorimetric data of the dyed fabric were determined by Color-Eye 7000A spectrophotometer (Gretag Macbeth, USA). The dye absorbance was measured in the visible spectrum region from 360 nm to 700 nm and the reflectance at the wavelength of maximum absorption ( $\lambda_{\max}$ ) was used to calculate the color yield of the dyed fabric by the Kubelka-Munk equation (1).

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

where  $K$  is the absorption coefficient of the substrate,  $S$  is the scattering coefficient of the substrate and  $R$  is the reflectance

of the dyed fabric at  $\lambda_{\max}$ .

Color fastness was evaluated according to the respective international standards: fastness to washing, ISO 105-C04 (2010); fastness to rubbing, ISO 105-X12(2001); fastness to perspiration, ISO 105/E04 (2013).

### Hydrolysis and High Pressure Liquid Chromatography (HPLC) Analysis of the Disperse Dyes

The hydrolysis of the disperse dyes was investigated under the alkaline condition, NaOH 5 g/l, using PYROTEC-2000 IR dyeing machine (Roaches International Ltd., UK) in the absence of PET fabric samples. The dispersive system was prepared at room temperature with water, 10 ml, the milled dye, 0.1 g, and NaOH, 0.05 g. Then heated to 130 °C and kept for 30 min and 60 min, respectively. The dye solution was cooled, and neutralized with acetic acid to pH 7. The dye crystal was washed with acetone, 10 ml, and the acetone solution was added to the cooled dye solution. The dye solutions were dried at 85 °C. The analysis of hydrolysis behavior of the dyes was carried out using HPLC (LC-10A TVP Liquid Chromatography, Shimadzu Corporation, Japan). A mixture of acetonitrile and water (80/20 v/v) was used as the mobile phase with the flow rate, 1 ml/min.

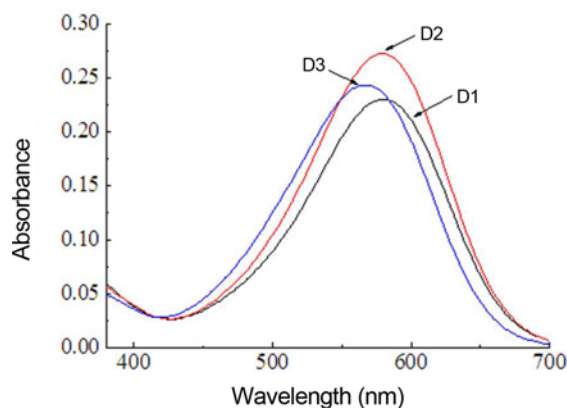
### Theoretical Calculation Using Density Functional Theory (DFT)

Gaussian 09 program package was used to optimize geometry and to investigate the synthesized azo dyes. Ground state geometry of the dyes in gas and solvent was optimized in their symmetry using DFT [20,21]. The basis set of B3LYP/6-31G (d) was used in all calculation.

## Results and Discussion

### Absorbance Spectra and Dyeing Properties under the Conventional Acid Condition

The UV-vis spectra of D1, D2, and D3 in acetone (AT) are shown in Figure 1. The concentrations of three dyes in AT were  $1 \times 10^{-5}$  mol/l, respectively. The maximum absorption



**Figure 1.** UV-Vis spectra of the disperse dyes (acetone as solvent).

**Table 1.** Spectrum properties of three disperse dyes in different solvents

Dyes	$\lambda_{\max}$ (nm)			$\epsilon \cdot 10^4$ (L mol <sup>-1</sup> cm <sup>-1</sup> )		
	DMF	AT	ACN	DMF	AT	ACN
D1	596	580	576	5.49	5.69	5.54
D2	594	580	577	6.36	6.46	6.38
D3	584	567	565	6.76	6.68	6.62

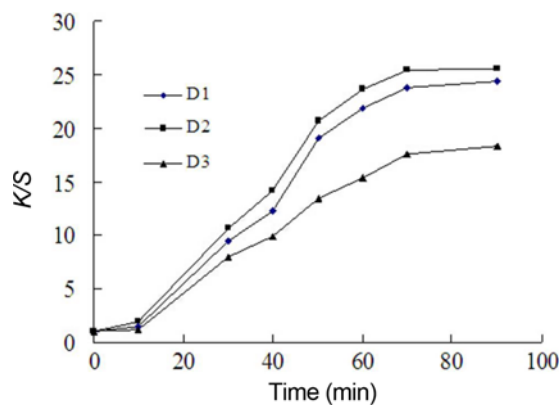
Note: DMF (dimethyl formamide), AT (acetone), ACN (acetonitrile).

peaks, at 580 nm for D1, 580 nm for D2, and 567 nm for D3, were assigned to the  $\pi$ - $\pi^*$  transition of the conjugated system. Compared with those of D1 and D2, the maximum absorption ( $\lambda_{\max}$ ) of D3 decreased 13 nm, because of the substituent effect of N-benzyl. The UV-vis spectra of them in the different solvents were measured and the data being summarized in Table 1.

The maximum absorptions of three dyes in DMF showed bathochromic shift compared with those of them in AT and ACN because of DMF's polarity. The molar extinction coefficients ( $\epsilon$ ) of D1 were slightly lower than those of D2 and D3 in all three solvents because of the effect of the hydroxyl polarity. Compared with those of D1 and D2, the maximum absorptions of D3 in three solvents decreased. It might be attributed to the stereochemistry effect of the N-substituted benzyl group.

### Dyeing Property of Three Dyes for PET under Acid Condition

The dyeing performances of three dyes under the conventional acid condition (pH 4.5) were carried out at the dye concentration 2.0 % (o.w.f.). *K/S* curves of the dyed fabrics are shown in Figure 2. *K/S* values of the dyed PET fabrics increased with the dyeing time. It indicates that PET fabrics were well dyed using three dyes under the acid condition. The color yields of D1 and D2 were higher than that of D3. Three synthesized dyes based on benzisothiazole

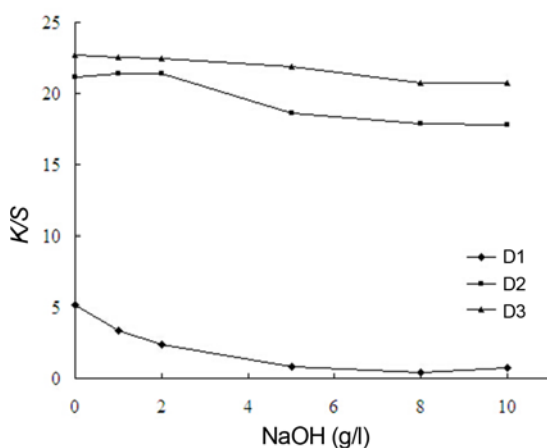


**Figure 2.** *K/S* curves of PET fabrics dyed with three dyes under the acid condition.

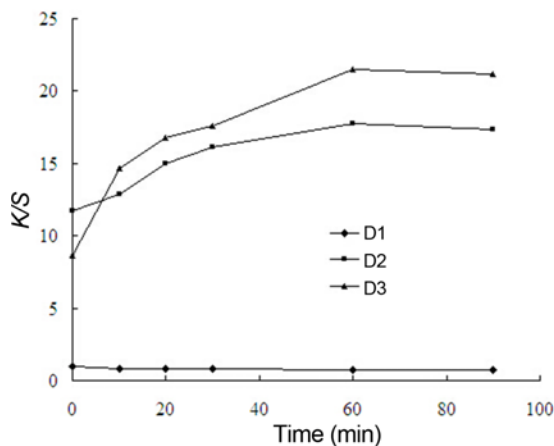
had excellent dyeing property for PET fabric under the acid condition.

### Dyeing Property of Three Dyes for PET under Alkali Condition

In order to investigate the resistant alkali property of three dyes containing cyano group, the dyeing procedure of The PET fabrics dyed with three dyes under the alkali condition, NaOH concentration, from 1 g/l to 10 g/l, were carried out at the dye concentration, 2.0 % (o.w.f.).  $K/S$  curves of the dyed fabrics with three dyes under the different NaOH concentrations are shown in Figure 3. With increasing NaOH concentration, from 1 g/l to 10 g/l, the color yields of D2 and D3 slightly decreased. The color yield of D3 for dyeing PET under the alkali condition had nearly no change. However, the color yield of D1 for dyeing PET under the alkali condition was the worst among three dyes. The samples using D1 almost could not be dyed. It indicates that D2 and D3 had excellent color yields under the alkali condition, whereas D1 was very



**Figure 3.**  $K/S$  curves of the disperse dyes under different NaOH concentrations.



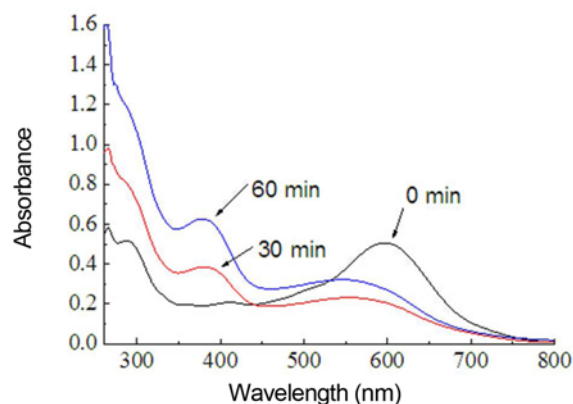
**Figure 4.**  $K/S$  curves of the dyed PET fabric under the alkali condition.

unstable.

The dyeing rates of three dyes for PET fabrics were also measured in the presence of NaOH, 5 g/l, and shown in Figure 4. The  $K/S$  values of D2 and D3 increased with the dyeing time. However, PET fabric could not be dyed using D1 when the concentration of NaOH was 5 g/l. This shows that D2 and D3 had the excellent resistant alkali stability. The levelling property of D2, and D3 were measured by color difference ( $\Delta E$ ) among five points on the same fabric, respectively. All the  $\Delta E$  measured are less than 0.5. The results indicated that the dyes possess good levelling properties on the PET fabrics. They could be applied to dye PET fabric under the alkali condition. Especially, the color yield of D3 for dyeing PET fabric under the alkali condition was the highest. The results were related to the intermolecular and intramolecular interaction of the dyes due to the different substituent effects.

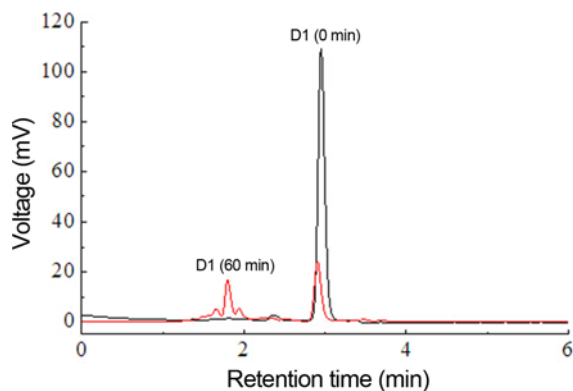
### Structure Analysis of the Resistant Alkali Stability

The structure differences of three heterocyclic azo disperse dyes containing cyano group based on benzisothiazole only lie in N-substituent tail group of the coupling component, the ethoxyl for D1, the ethyl for D2, the benzyl for D3. The UV-vis spectrum and HPLC analysis were used to analyze the stability of them. The dyes, D1 and D2, were treated for a certain time at NaOH (5 g/l) solution, 130 °C, respectively. After cooling, acetone, 20 ml, was added to the dissolved dye. Then the cooling solution was dried at 85 °C. The solid residual product was applied to the component analysis. The absorbance spectra of the dyeing residual product of D1 are shown in Figure 5. It indicates that the dye residual component changed after treating 30 min and 60 min at NaOH, 5 g/l. The component at  $\lambda_{\max}$  580 nm decreased with time. Another component at  $\lambda_{\max}$  390 nm increased, which was yellow-brown product. The decomposition might take place when D1 was in the alkali solution under 130 °C. The results of HPLC analysis are shown in Figure 6. There was a main peak of D1 without the treatment at the retention time 2.957 min. After treating 60 min, the main peak at the retention

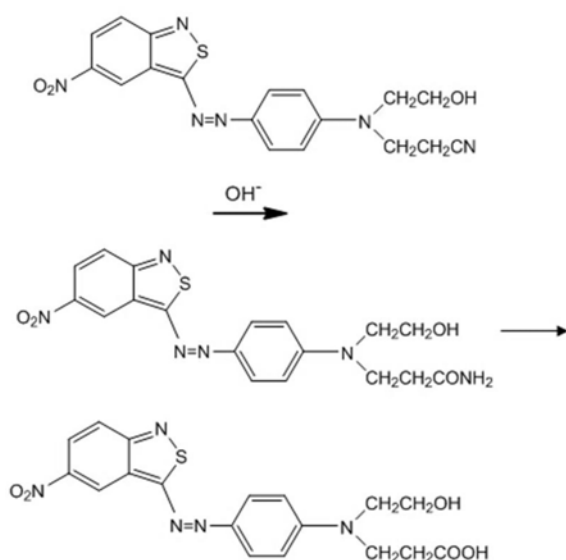


**Figure 5.** Absorbance spectrum of the dyeing residual of D1.

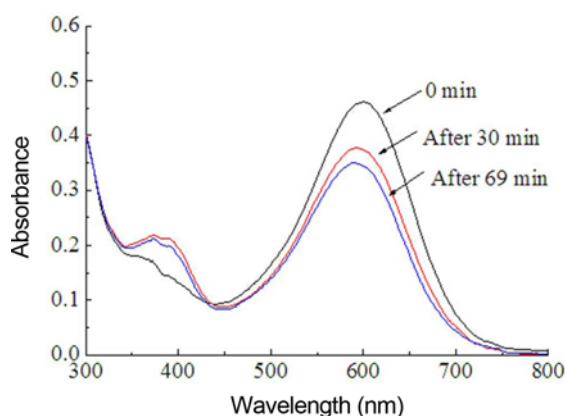
time 2.957 min sharply decreased. Another peak at the retention time 1.841 min appeared. The component should be the cyano group hydrolyzed product at the alkali



**Figure 6.** HPLC analysis of the dyeing residual product of D1.



**Scheme 2.** Hydrolyzing mechanism of D1.



**Figure 7.** Absorbance spectrum of the dyeing residual of D2.

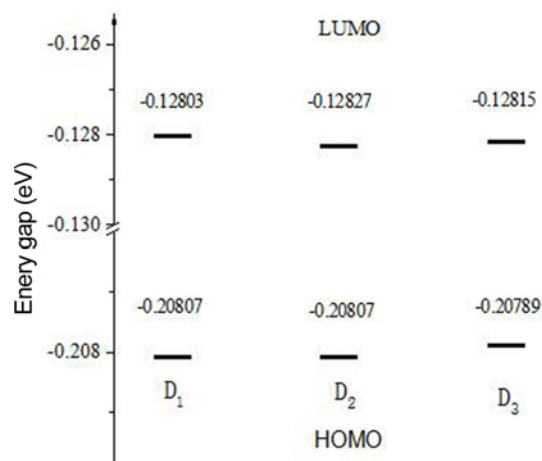
condition. The cyano group stability of D1 should be related to form the hydrogen bond with the adjacent ethoxyl group and water molecules. The hydrolyzing mechanism of D1 is shown in Scheme 2.

D2 was treated by the similar method in the NaOH (5 g/l) solution, at 130 °C. The absorbance spectra of residual product of D2 are shown in Figure 7. The dye residual component had not obvious change after treating 30 min and 60 min at the alkali condition. The chemical structure of D2 was stable at the alkali condition.

### Theoretical Calculation of Frontier Molecular Orbital and Solvation Energies

In order to understand the surprising phenomenon, DFT calculations have been carried out to optimize the structure of the dye molecules. All calculations were performed at the level of B3LYP/6-31G (d) in Gaussian 09 program. The different frontier molecular orbital was investigated to understand the electronic transition and charge delocalization within the push-pull chromophores. The comparative increase and decrease in the energy of the occupied (HOMO's) and virtual orbitals (LUMO's) gives a qualitative idea of the excitation property. The energy level diagram represents the alignment of various energy levels and is shown in Figure 8. The HOMO energy levels of the dyes were similar and were not obviously affected by the introduction of tail groups. However, their LUMO energy levels had slightly difference. Due to the effect of the electron-withdraw of the hydroxyl group, the energy gap of D1 was slightly higher than those of D2 and D3. The delocalization of  $\pi$  electron within the push-pull chromophore of D1 was certain restricted. It is also explained that the molar extinction coefficient ( $\epsilon$ ) of D1 was slightly lower than those of D2 and D3 because of the restricted delocalization of  $\pi$  electron in the dye molecule.

The ground state geometries of the dyes, D1, D2 and D3 in



**Figure 8.** Energy level diagram depicting the HOMO and LUMO levels of the dyes.

**Table 2.** Solvation energies of the dyes in gas and solvent

Compound	D1	D2	D3
Gas phase (Hartree)	-1647.78	-1572.57	-1764.31
Water (Hartree)	-1647.82	-1572.60	-1764.36
Solvation (kcal/mol)	23.30	17.49	18.02

gas and solvent were optimized in their symmetry using DFT. The solvation energies of them in water were calculated and listed in Table 2.

The solvation energy of D1 in water was obviously higher than those of D2 and D3. It can be explained that the hydrogen bond was easy to be formed between water molecule and hydroxyl of D1 as well as the adjacent electron-withdrawing cyano group. The formation of hydrogen bond affected the dipole moment of cyano group in the dye molecule. So, the electron-withdrawing cyano group of D1 was easy to be hydrolyzed under the alkaline condition. The solvation energy of D2, and D3 in water was obviously lower and could form different stereochemical structures. In our previous research [14], the single crystal of the similar structure with D2, D3, 3-(3-methyl-4-*N*-ethyl-*N*-benzyl-phenyldiazenyl)-5-nitro-2,1-benzisothiazole, was analyzed. It indicates that two planes of the  $\pi$ - $\pi$  conjugated system of two adjacent dye molecules were all parallel and formed the triclinic crystal. There were the  $\pi$ - $\pi$  stacking interactions between the conjugated systems of two adjacent dye molecules. Compared with D1, the cyano group of D2 or D3 cannot form the hydrogen bond with hydroxyl. At the same time, the  $\pi$ - $\pi$  stacking interaction between the adjacent two dye molecules benefits to protect the cyano group of the dyes from hydrolysis. So, D2 and D3 have good stability.

### Fastness Property of the Dyed PET Fabric

Color fastnesses of the dyed fabric with three dyes according to the standard shade were also examined. The fastness properties of the dyed PET fabrics with D2 and D3 at the alkali condition were excellent and almost the same with those of the dyed PET fabrics with the conventional acid method.

### Conclusion

Three dyes containing cyano group based on benzisothiazole were synthesized. D2 and D3 had excellent color yields under the alkali condition, NaOH concentration from 1 g/l to 10 g/l. However, D1 was very unstable and took place of decomposition under the alkali condition. The instability of D1 is closely related to the hydroxyl and cyano group of the dye tail. The solvation energy of D1 in water was obviously higher than those of D2, and D3. The electron-withdrawing effect of the hydroxyl affected the energy gap of HOMO and LUMO orbitals. D2 and D3 showed good stability in the

strong alkali medium. The fastness properties of the dyed PET fabrics with D2 and D3 at the alkali condition were also excellent.

### Acknowledgment

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