Studies on UV-Visible, Fluorescent Spectral Properties and Solvatochromic behavior of Naphthalimide Compound Containing Quaternary Ammonium

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ABSTRACT

*T*his paper presents the results of absorption spectra, fluorescence properties and the effect of solvents on UV-Vis spectra of dye quaternized 4-acetylamino-N-2-aminomethylpyridine-1,8-naphtha-limide. The fluorescence properties of the dye were evaluated. It's Stokes shift value was 6140 cm^{-1} in DMF. The solvatochromism behavior of the novel compound is investigated by studying its spectra in pure organic solvents of different characteristics. The factors that affect solvatochromism including solvent polarity are discussed. Finally, the fluorescent dye was applied on polyamide fabrics in order to investigate its dyeing properties. Wash, light, and rubbing fastnesses of the dyed fabrics were measured by standard methods. Moreover, the build-up properties of the synthesized dye were measured on polyamide fabrics. Dyed polyamides had very good wash and rubbing fastness degrees and moderate light fastness. The antibacterial activity of the dyed fabrics was evaluated. *Prog. Color Colorants Coat. 10 (2017), 163-172© Institute for Color Science and Technology.*

1. Introduction

Organic fluorescent dyes have a wide range of applications in paint and coatings, cosmetics, food, inks and textile industries [1, 2]. By far many chemical structures of organic fluorescent dyes were synthesized and their properties studied. One group of fluorescent dyes is 1,8-naphthalimide derivatives which used as yellow daylight fluorescent compounds, fluorescent dichroic dyes in liquid crystal displays and fluorescent brighteners in detergents, textiles, papers, plastics etc [1-6].1,8-Naphthalimide derivatives with amino groups in the C-4 position have a bright yellow color and show very intense yellow-green fluorescence properties and with acetylamino groups have a fluorescent blue color.

In many cases, 1,8-Naphthalimide dyes are used as solutions and thus their optical properties are influenced by the solvent environment. Therefore, the investigation of their behavior in various solvents is of interest [7–12]. The dye molecules can undergo various interactions with solvent molecules thereby changing their spectral behavior. Absorption spectra are the most versatile and simple means of studying such transitions in molecules. A shift in absorption wavelength in various solvents reveals information about the solute-solvent interactions [13]. This shift is usually termed as solvatochromism and depends on the electronic structure of the sample and solvent molecules, which in turn determines the intermolecular solute-solvent interactions in the ground and the first excited state. The solvatochromic effect refers to a strong dependence of absorption and emission spectra with the solvent polarity [14, 15]. Since polarities of the ground and excited state of a chromophore are different, a change in the solvent polarity will lead to

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differential stabilization of the ground and excited states and thus, a change in the energy gap between these electronic states. Therefore, variations in the position, shape, and intensity of the absorption spectra can be a direct measure of the specific interactions between the solute and solvent molecules. Solvatochromism can show both negative and positive values [16]. The sign of the solvatochromism depends on the difference in dipole moment between the ground and excited states of the chromophore. If the ground state is more stabilized than the excited state due to solvation by solvents of increasing polarity, negative solvatochromism is displayed. Negative solvato-chromism corresponds to hypsochromic shift, whereas, positive solvatochromism corresponds to bathochromic shift with increasing solvent polarity [17].

The compounds containing quaternary ammonium salts play an important role in many antimicrobial systems [18]. There are several methods to confer antimicrobial properties onto textile materials but it has been suggested that using dyes with color properties and antimicrobial properties leads to the most satisfactory outcomes [19]. However, there are few papers about the investigation of the spectrophoto-metric, antimicrobial and dyeing properties of fluorescent naphthalimide dyes and dyed fabrics.

In this study we have investigated spectrophoto-metric and dyeing properties, antibacterial and antifungal activities of a fluorescent cationic dye on polyamide fabrics.

2. Experimental

2.1. Materials and apparatus

All compounds used in this study were of analytical grade unless otherwise stated. The final dye and its intermediates were used as DMF solution with five different concentrations, i.e. 1×10^{-4} M, 2×10^{-4} M, 4×10^{-4} M, 6×10^{-4}M and 8×10^{-4} M. The UV–visible spectra of dye were measured with a Cecil 9200 double beam spectrophotometer. λ_{max}, ε and solvatochromic effect of dye have been determined. Fluorescence spectra of dye solutions were evaluated by Osean Optics Usb2000flg Fluorometer. Fluorescence quantum yield was determined on the basis of the absorption and fluorescence spectra, using fluorescein as standard (Φ_{ref} = 0.95). Polyamide 6 fabrics (Alyaf Co., Iran) were utilized throughout the study.

2.2. Dyeing procedure

Polyamide fabrics were dyed with fluorescent dye at pH 5 using a solution produced from acetic acid (1%) and dispersing agent (1%) mixture, the procedure which is recommended by dye manufacturer for dyeing at 40 ˚C as a starting point. All dyeing trials were performed using a rapid laboratory dyeing machine at L:G 100:1. The fabrics were washed for 30 min in the bath of nonionic detergent (5 g/L) at 80 ˚C. The concentrations of the dye dispersions were 0.4, 0.8, 1.2, 2, 4 and 6 g/L. The temperature of dye bath was then gradually raised from 40 ˚C to 100 ˚C at rate of 2 ˚C/min for 45 min. Dyeing was continued for 60 min at this temperature. After completion of dyeing cycle, the fabrics were rinsed with hot and then cold water and then air dried. Effects of operational parameters such as concentration on the grafting yield were monitored by changes in dye up-take ability of substrates as expressed by color strength (K/S) values. K/S values were calculated at λ_{max} using Kubelka–Munk equation (Eq. (1)) as follows:

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

where R is the reflectance, K and S are the absorbance and scattering coefficients, respectively.

2.3. Fastness properties

Wash fastness was measured by the standard ISO105-A02:1993 method. The washing was conducted for 30 min at 60 ˚C, rinsed with cold water, air dried, and analyzed with gray scale. Light fastness test BS 1006-1990 UK-TN was evaluated with the xenon arc lamp using blue reference samples. The rub fastness test was performed according to ISO 105-X12:1993(E) standard using a crockmeter. For the wet rub test, the testing squares were thoroughly immersed in distilled water; the rest of the procedure was the same as in the dry test. The staining on the white test cloth was assessed according to the gray scale.

2.4. Antimicrobial activity of the treated fabrics

Antimicrobial properties of treated fabrics were examined according to the AATCC test method 100–1999, a quantitative antimicrobial test method performed under dynamic contact conditions using test bacteria of *E. coli* and *S. aureus*. In this method, a

number of test tubes, each containing 5.0 mL of Muller-Hinton broth (MHB, Difco, England) was autoclaved for 20 min at 121˚C. Bacterial inoculums (1.0 ± 0.1 mL) were added to the circular fabric swatches (1.0 g). These inoculums were nutrient broth cultures containing 10^6–10^7 mL^{-1} CFU (colony-forming units) of bacteria. Positive control tubes contained 5.0 mL of the nutrient broth medium with tested bacterial concentrations of 10^5–10^6 CFU/mL while negative control tubes contained only the inoculated broth. Tubes were incubated at 37 ˚C for 24 h at a constant temperature of incubator. Treated nylon fabrics with dye were cut into 25 ×50 mm and used for antimicrobial tests. All fabrics were incubated for 24 h at 37 ˚C. Then, 100 mL of solution was taken from each incubated fabric, and distributed over an agar plate. All plates were incubated again for 24 h and the colonies formed on them were counted [19].

The antimicrobial activity was expressed in terms of reduction of the organisms (%) after contacting test specimen compared to the number of bacterial cells surviving after contacting the control. The percentage reduction was calculated using Eq. (2) as follows:

$$R = (A-B/A) \times 100 \% \qquad (2)$$

where R is the percentage reduction of the bacterium; A, the number of bacterial colonies from untreated fabric, and B, the number of bacterial colonies from treated fabrics. The test was repeated three times and the results were reported as mean percentage reduction of the bacterium.

3. Results and Discussion

3.1. Photophysical properties

The synthesis of cationic dye has been reported in our previous work [20]. The chemical structures of synthesized dye are illustrated in Figure 1. Photophysical properties of organic materials are important in order to recognize their functioning in device applications. The photophysical properties of synthesized dye are generally dependent on the polarization of the 1,8-naphthalimide backbone. Generally, light absorption in these dyes generate charge transfer, which leads to D–π–A interaction between the substituents at the C-4 position and carbonyl groups of the naphthalimides and may be influenced by environment of the media [21–24].

3.1.1. Absorption spectral properties

The nature of the N-substitutions and C-4 position substitutions have important role in the preparation of fluorescent dyes derived from naphthalimides and as having pronounced effects on the color of the dyes such as position of absorption maximum (λ_{max}) and brightness of shade.
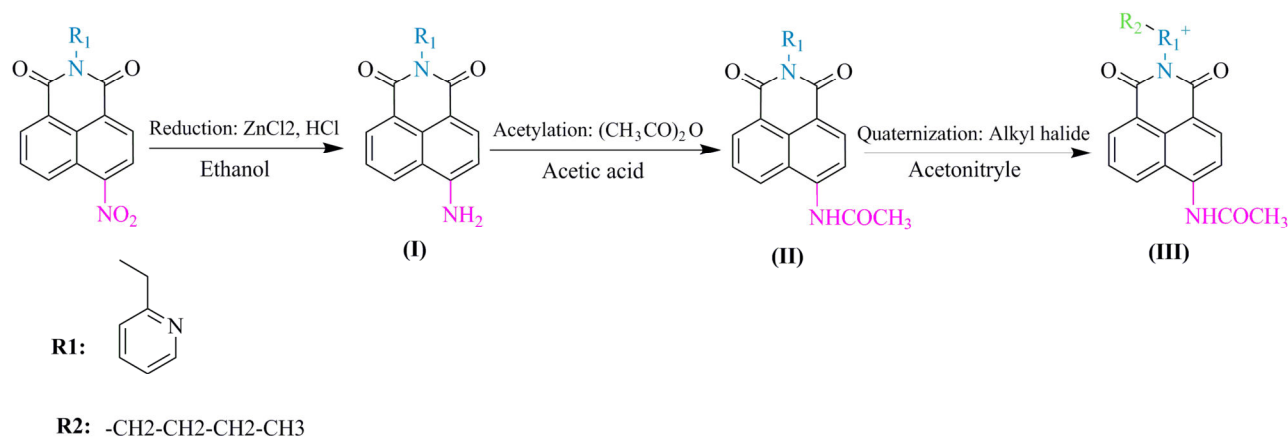

Figure 1: The synthesis pathway of the naphthalimide dyes.

Therefore, the wavelengths of maximum absorption and molar extinction coefficients are important parameters for dyes. A higher extinction coefficient and a broader half-band width are attributed to a stronger chromophore which makes the final product more cost effective [25, 26]. The UV-Visible absorption data for cationic dye and other prepared dyes were determined in toluene, acetone, ethanol, tetrahydrofuran and DMF as solvent (Table 1).

The molar extinction coefficients of the cationic dye and other prepared dyes in DMF are in the range of 1.02×10^4 to 1.9×10^4 $\text{lmol}^{-1}\text{cm}^{-1}$. The molar absorption coefficient of the dye was increased by the maximum absorption wavelength. For the synthesized dye and its intermediates, molar absorption coefficient values tend to rise with increasing electron withdrawing capacity in the acceptor ring and increasing electron donating capacity in C-4 position 1,8-naphthalimide ring. In this respect, the presence of carbonyl groups in naphthalimide ring and acetylamino and amino electron donating groups in C-4 position 1,8-naphthalimide ring can increase molar absorption [27].

The molar extinction coefficient of final dye (**III**) was lower than that of its intermediates (**I** and **II**) because of a quaternary ammonium butyl group present in the structure of dye **III**. Quaternary ammonium butyl group decrease color intensity of final dye.

The final dye and other prepared dyes had hues from blue to yellow as illustrated in Table 1 and the UV-Vis absorption spectra of prepared dyes are shown in Figure 2. The final dye and other prepared dyes have λ_{max} between 370-432 nm (Table 1).

3.1.2. Fluorescent properties

1,8-naphthalimides can show strong fluorescence effect only when an electron-donating group is substituted on the naphthalimide ring, usually at the C-4 or C-5 position. The fluorescent characteristics of final dye and its intermediates such as fluorescence maxima (λ_F), Stokes shifts ($\nu_A - \nu_F$), oscillator strengths (f), quantum yields of fluorescence (Φ_F) and energy yields (E_F) were measured in DMF and are represented in Table 1. In DMF solutions, novel dye **III** show intense blue fluorescence due to the charge transfer in the naphthalimide compound from the electron donating acetyl amino group at C-4 position to the accepting carbonyl groups. The emission of dyes is in the range of about 481–524 nm (Figure 3). In this research, it could be concluded that the presence of electron amino donating groups in C-4 position naphthalimide derivatives result in a red shift (Dye **I**). Dyes **II** and **III** consisting of acetyl amino groups have blue shifts due to their weak electron donating characteristics.
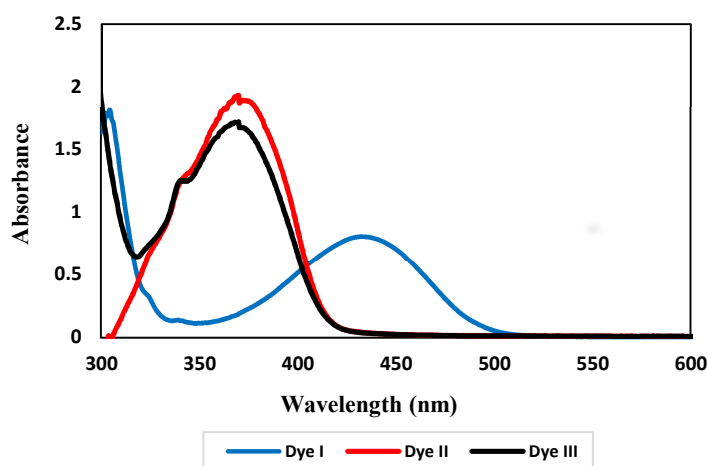

Figure 2: The absorption spectra of dye (III) and its intermediates (I & II) in DMF.

The values of the oscillator strength (f) are a true measure of the color intensity for the investigated final dye. They show the effective number of electrons whose transition from the ground to the excited state gives the absorption area in the electron spectrum. The oscillator strength was calculated according to the following equation 3 [28]:

$$f = 4.32 \times 10^{-9} \Delta \nu_{1/2} \varepsilon_{max} \qquad (3)$$

where $\Delta\nu_{1/2}$ is the width of the absorption band in cm^{-1} at $\varepsilon_{max}/2$. The values of f are 0.42 for novel dye and 0.37 and 0.52 for its intermediates **I** and **II**, respectively. Stokes shift was calculated by the following expression (equation 4).

$$(\nu_A - \nu_F) = (\frac{1}{\lambda_A} - \frac{1}{\lambda_F}) \times 10^7 \qquad (4)$$

Stokes shift of novel dye and its intermediates was between 4070 and 6140 cm^{-1} (Table 1). It is due to the energy losses during the transition to the excited state. This is an evidence of notable steric changes which the molecules undergo during the transition from the basic to the excited state.

The fluorescence quantum yield of a sample Φ_F is defined as the fraction of photons emitted per number of absorbed photons. The quantum yield is often calculated by comparing a sample with a reference substance of known quantum yield Φ_{ref} such that equation 5:

$$\Phi_F = \Phi_{ref}(\frac{S_{sample}}{S_{ref}})(\frac{A_{ref}}{A_{sample}})(\frac{n^2_{sample}}{n^2_{ref}}) \qquad (5)$$

where n_{sample} (n_{ref}) is the refractive index of the sample (reference), S_{sample} (S_{ref}) is the integrated emission of the sample (reference), and A_{sample} (A_{ref}) is the absorbance of the sample (reference) at the excitation wavelength [28]. The quantum fluorescence yield of the dyes **I-III** measured in DMF solution is very good ($\Phi_F = 0.37–0.26$).

The energy yield of fluorescence (E_F) could also be utilized to characterize the fluorescence efficiency of fluorescent dyes which is calculated by equation 6 [29]. The value of the energy yield of fluorescence of novel dye was in the region of 0.2.

$$E_F = \Phi_F \frac{\lambda_A}{\lambda_F} \qquad (6)$$

Table 1: Absorption and fluorescent characteristics of dye and its intermediates in DMF solution.

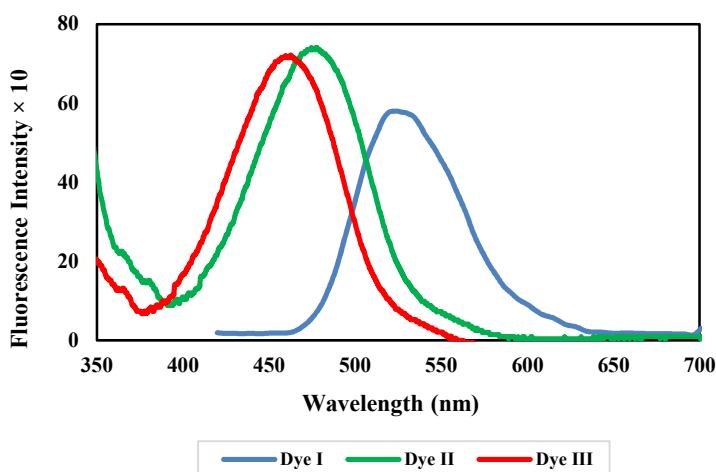| Compound	λ_A (nm)	λ_f (nm)	ε (lmol^{-1}cm^{-1})	ν_A-ν_B (cm^{-1})	Φ_F	f	E_F
I	432	524	1.9×10^4	4070	0.37	0.37	0.31
II	374	482	1.2×10^4	5990	0.27	0.52	0.21
III	370	479	1.0×10^4	6140	0.26	0.42	0.2

Figure 3: The emission spectra of dyes I-III in DMF.

3.1.3. Solvatochromic Effect

Since the tautomeric equilibria strongly depend on the nature of the media [30]. The solvent polarity influences the characteristics of the spectra of the dye molecules, the phenomenon which is called solvatochromism [30]. From idealized theories, the solvent dielectric constant is often predicted to serve as a quantitative measure of solvent polarity. The higher the dielectric constant of a solvent, the more polar it is. The behavior of selected novel naphthalimide dye in five protic and aprotic solvents was studied at room temperature. For this purpose, the absorption spectra of the new dye at the concentration of 8×10^{-4} mol L^{-1} were recorded over the λ range between 300 and 700 nm in the selected solvent set. The λ_{max} shift of dye **III** in various solvents is shown in Figure 4. We found that the absorption band at 346-370 nm generally shows bathochromic shift (positive solvatochromism) as the polarity of solvent was increased (Table 2). The influence of solvents for the prepared dye increases in the order DMF > CH$_3$CH$_2$OH > acetone > THF > toluene. As it can be seen, the absorption spectra of the dye in DMF solution are red shifted as compared to the dye spectra in other solvents, indicating relatively strong interaction between the dye molecules and the DMF environment. Similar results have also been observed by other researchers [31-33].

3.1.4. Solvatofluorochromism Effect

In order to study the effects of solvents on the fluorescent properties, fluorescence intensities of final dye in different solutions of the same concentration were detected in DMF, acetone, CH$_3$CH$_2$OH, THF and toluene, respectively, and the results are shown in Table 2. The wavelength shifted red with the enhancement of solvent polarity. The wavelength in the stronger polar solvent DMF was at 479 nm, which shifted 16 nm and 17 nm compared to those in CH$_3$CH$_2$OH and acetone, respectively. The conjugated electron density was lowered and a strong intermolecular charge transfer occurred with the increase of solvent polarity, which resulted in the increment of fluorescence spectra. These results were also obtained in other researches [34, 35]. The behavior can be explained by π–π* electron transfer on S0–S1 transition providing the long-wavelength bands in the UV region. On excitation of the molecule, the dipole moments and the polarity of the C=C bonds tend to be larger wavelength bands than in the ground state. Molecules in more polar solvents are more stable because of smaller internal interactions in the excited state and a smaller difference in energy between the excited state and the ground state. This accounts for the observed bathochromic shifts [36].
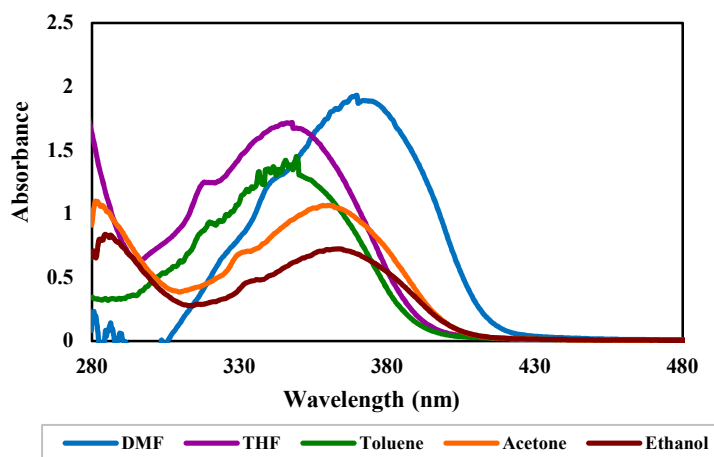

Figure 4: The UV–Vis absorption spectra of the dye III in various solvents.

3.2. Dyeing properties

3.2.1. Effect of dye concentration

In general, color strength of synthesized dye on polyamide fabrics is good because the cationic dyes can be connected via ionic bonding to the polyamide fabric. The impact of dye concentration on the dye-ability of polyamide fabric was investigated. K/S values as a function of dye concentrations are shown in Figure 5. It is observed that the increase of concentration of dye on fabrics was resulted in an increase in K/S values apart from the type of dye used. Moreover, it seems that 3 g/L of dye was quite effective in surface alteration of nylon substrate. Indeed, K/S values increased up to around 3 g/L fluorescent dye where reached plateau. No appreciable improvement in K/S values was obtained with further raise in dye concentrations higher than

3 g/L, suggesting the saturation of polyamide surface beyond this concentration.

3.2.2. Fastness properties

Textiles are exposed to repeated washing and rubbing during their usage. Hence, durability of the finish applied on fabrics at these conditions is very important [37]. The results of wash, light, and rubbing fastness of the fluorescent dye on polyamide fabrics are presented in Table 3. According to the results, wash fastness ratings for staining of adjoining fabrics are very good and those for color change are also in acceptable level. Rubbing fastness of the dyed samples measured in dry and wet conditions is in good level. The light fastness of the dyed fabrics is moderate as well.

Table 2: Spectral Data (absorption & emission) of the Synthesized Dye in Various Solutions.

Solvents	Dielectric Constant	λ_A (nm)	λ_f (nm)
Toluene	2.38	346	450
THF	7.58	355	455
Acetone	20.7	360	462
Ethanol	24.5	362	463
DMF	36.7	370	479

Table 3: Color Fastness of Dyed Polyamide Fabrics.

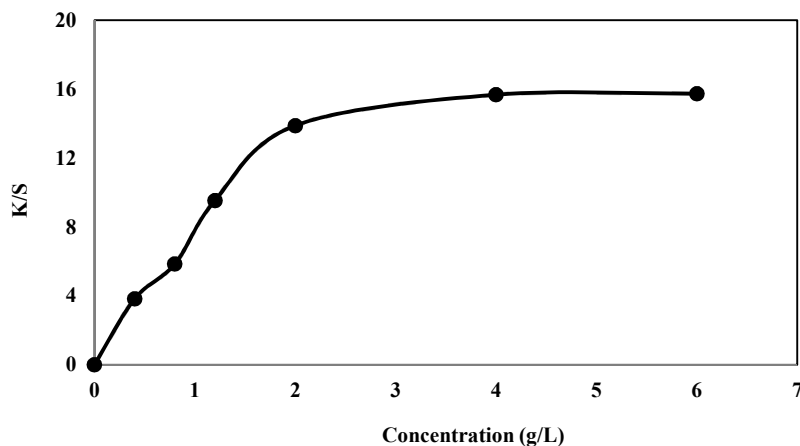| Dye	Washing Fastness			Light Fastness	Rubbing Fastness	
	Staining		Change		Wet	Dry
	Polyamide	Cotton	Polyamide			
III	4-5	5	4-5	4	4-5	4-5

Figure 5: Build up curves for the cationic fluorescent dye

Table 4: Antibacterial Efficacy of the Dyed polyamides in two Concentrations.

Dye	Bacteria	Dyes concentration (g/L)	A	B	R (%) =(A-B) /A × 100%
III	*E. coli*	2	2×10^5	1.36×10^4	93.20
		4	2×10^5	9.86×10^3	95.07
	S.aureus	2	5.4×10^5	1.98×10^4	96.33
		4	5.4×10^5	1.08×10^4	98.00

3.3. Antibacterial properties of dyed fabrics

In this study, antibacterial properties of the fabrics were investigated according to AATCC test method 100–1999 against two bacteria *E. coli* and *S. aureus* and the results are showed in Table 4. The results clearly showed strong microbial reduction in cases of *E. coli* and *S. aureus* bacteria. The most accepted mechanism for microbial inhibition by fluorescent dye may be the interaction of the positively charged amine groups ($-NH_2$) with the negatively charged residues at the cell surface of many microorganisms, which causes extensive alteration of cell surface and cell permeability. It can be observed that with an increase of dyeing concentrations, the antimicrobial activities of fabrics also increase in 1 h of contact time.

4. Conclusion

In this work, the spectrophotometric properties of a new dye in DMF solvent were examined and the absorption maxima, fluorescence emission and their intensities have also been obtained. The photophysical properties revealed that prepared dye is fluorescent and absorption spectrum was 370 nm while fluorescence spectrum was 479 nm with good quantum emission yield. Absorption spectra of the dye are directly dependent on the solvent. It was observed that the λ_{max} value of dye showed bathochromic shift. Prepared dye showed positive solvatochromic behavior as the polarity of solvent was increased. It is capable of dyeing polyamide fabrics with very good wash and good rubbing fastness degrees. Finally, antibacterial activity of dyed fabrics was determined according to the AATCC 100 method. All the treated fabrics exhibited antimicrobial efficacy against *E. coli* and *S. aureus* bacteria.

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