



## Research Article

# Synthesis, characterization and application of novel 1, 3-bis[(furan-2-yl)methylene]thiourea functional dye on wool and cotton fabrics

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## Abstract

Novel 1,3-bis[(furan-2-yl)methylene]thiourea (BFMT) dye was synthesized from condensation reaction between sugarcane bagasse derived furfural and thiourea. The synthesized BFMT dye was characterized by physicochemical (melting point and CHN) and spectrometry (UV–Vis, FTIR, <sup>1</sup>H-NMR and <sup>13</sup>C-NMR) analyses. The synthesized BFMT dye was screened for its antimicrobial (antibiotic and antifungal) properties. BFMT dye was applied on wool and cotton fabrics. The functional properties of BFMT dyed fabrics (color value, color strength, fastness properties, antimicrobial activities, ultraviolet protection factor (UPF), and mechanical properties) were assessed. Chemical composition of synthesized BFMT dye was determined by CHN analyzer, while its structure was confirmed by FTIR and NMR spectrometry analyses. BFMT dye solution showed reddish brown complementary color with  $\lambda_{\text{max}}$  485 nm on UV–Visible spectrophotometer. The antibiotic property of the synthesized dye was moderate, while antifungal property was excellent on tested microorganisms. BFMT dyed wool and cotton fabrics displayed good mechanical properties, air permeability, water vapor permeability and wettability. Dyed fabrics possessed excellent exhaustion (> 80%), fastness properties, ultraviolet protection factor (UPF > 40) and fungal growth inhibition rate (> 70%).

**Keywords** BFMT dye · Functional properties · Furfural · Spectrometry · Ultraviolet protection factor

## 1 Introduction

Textile apparels (clothing materials) are conventionally used for covering human body since pre-historical time [1]. These days, people demand for functional textile materials with attributes like attractive color for religious, social and comfort purposes [2]. Others are antimicrobial, resilience, crease free and ultraviolet light protection for medical and sport wears, flame retardancy and high mechanical strength for fire fighter garment [3]. Textile materials were first colored with natural dyes during Egyptians' civilization era. Egyptians later had tyran purple dyed silk, cotton and wool fabrics as customary symbol for royalty [4]. Ancient writers attributed antimicrobial and ultraviolet

protective properties, besides attractive color to natural dyes extracted from *Bridelia feruginea*, *Moringa oleifera*, *Acacia nilotica*, *Terminalia chebula*, *Quercus robur*, *curcuma longa*, *Bixa orellana* etc. [5]. Other merits of natural dyes to textile industry, consumers and environment are availability of precursors, biodegradability, safe handling and eco-friendly nature [6]. Food, cosmetics, drinks, photography, medicines and leather industries are some of other areas where natural dyes were used [7]. Low yield, Poor fastness properties, high cost, lack of uniformity and irreproducibility are some of demerits faced by natural dye commercialization [8].

In nineteenth century, discovery of synthetic dyes took glory of natural dyes away due to their cost effectiveness,

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high reproducibility, high yield and availability. Other qualities are wide range of shade, good uniformity and excellent fastness properties on substrates [9]. Therefore, synthesis of bio-based dye from agro-waste materials is a better technology of obtaining dyes with superior qualities. According to Abu-Dief and Mohamed [10], these bio-based synthetic (Schiff base) dyes are cost and time efficient with high affinity for substrates, high reproducibility, good uniformity, excellent fastness properties and wide range of brighter shades in comparison to natural dyes. Literature reported that Schiff base (bio-based synthetic) dyes possess antibiotic, antiviral, antifungal, antioxidant, anticancer, anti-malarial, anti-inflammatory and ultraviolet protective properties [11]. Cardamone [12] identified textile materials as potential media for microbial growth. Offensive effects; such as discoloration, deterioration, dermal infection and unpleasant odour are caused by microbial growth to cloths and wearers according to Callewaert et al. [13]. Application of bio-based synthetic dyes on textile materials (wool, cotton, silk and blended fabrics) is a recent technology of correcting natural demerit ability of these materials as potential media for promoting microbial growth according to Abu-Dief and Mohamed [10]. Dubrovski and Golob [14] categorized UV radiation into UV-A radiation (380–315 nm) capable of initiating premature ageing and skin wrinkling, UV-B (315–280 nm) capable of originating sun burn, skin cancers and cataracts and UV-C ( $\leq 280$  nm) is most dangerous but arrested by ozone layer. Synthetic bio-based dye application on textile apparel can protect human body from the effects of harmful UV radiation from sunlight due to presence of UV arresting components in them according to Korać and Khambholja [15]. Apart from their textile application, bio-based synthetic dyes can also be used in leather coloration, medicines, handicraft items and toys industry [10]. These attributes will create substantial market for BFMT dyed textile materials according to Abu-Dief and Mohamed [10].

The aim of present study was to synthesize 1, 3-bis[(furan-2-yl)methylene]thiourea (Schiff base) dye by condensation reaction, characterize it using physicochemical/ spectrometry analyses and assess its dyed fabrics for color strength, fastness properties (wash, rub, perspiration and light fastness), antimicrobial activity, ultraviolet protection factor (UPF) and mechanical property.

## 2 Materials and methods

### 2.1 Material

Thiourea, ethanol, methanol, acetone, chloroform deuterated dimethylsulfoxide (DMSO), n-hexane, benzene, glacial

acetic acid, sulfuric acid and sodium chloride were obtained from British Drug House Chemical Ltd (BDH). Sugarcane bagasse and white fabrics (cotton and wool) were obtained from Orisunmibare market, Osogbo, Osun State, Nigeria. Ten mice were obtained from animal house of Biochemistry Department, Federal University of Technology, Akure.

### 2.2 Methodology

#### 2.2.1 Preparation of furfural (precursor)

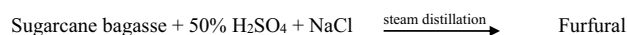
Sugarcane bagasse (SB) was washed with distilled water to get rid of dust and other related dirt. It was oven then dried for 3 h at temperature of 105 °C and ground to 1 mm mesh size. Furfural was obtained from prepared sugarcane bagasse by steam distillation according to Barbosa et al. [16]. Accurately weighed pulverized SB (15 g) was poured in a round bottom flask (500 mL) containing 50% H<sub>2</sub>SO<sub>4</sub> (150 mL) and NaCl salt (5 g) and distilled for 40 min to obtain furfural (Scheme 1).

#### 2.2.2 Synthesis of BFMT dye

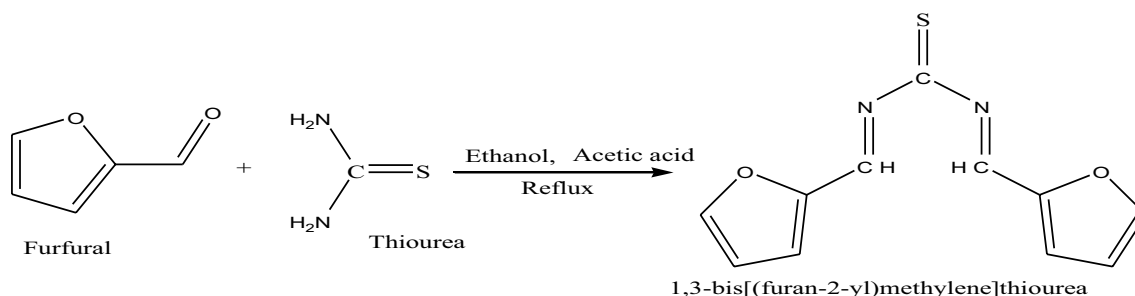
The novel 1, 3-Bis[(furan-2-yl)methylene]thiourea (BFMT) dye was synthesized by condensation reaction between bio-based furfural and thiourea according to Abu-Dief and Mohamed [10]. Ethanol (5 mL) dissolved furfural (10 mg) was reacted with thiourea (6.25 g) in presence glacial acetic acid (2 drops) as catalyst and refluxed for 1 h in round bottom flask (250 mL). After reaction period of 60 min, the reacting mixture was poured onto cold distilled water (250 mL) in a beaker (1 L) for BFMT dye to precipitate out within 15 min (Scheme 2). The precipitated BFMT dye was washed with distilled water, filtered by Watman filter paper and re-crystallized in ethanol within 15 min [17]. The purity of synthesized BFMT dye was confirmed by thin layer chromatographic technique using silica gel coated plate.

#### 2.2.3 Characterization of BFMT dye

The synthesized BFMT dye was characterized by physicochemical and spectrometry analyses. The melting point was determined using capillary melting point technique, solubility property was determined with various solvents (organic and aqueous) and color was detected with naked eyes. Elemental analysis was carried out according to Gehre and Strauch [18]. Homogenized BFMT dye (~0.1 g) was loaded into CHN analyzer (Perkin Elmer 2400) operated at



**Scheme 1:** Production of furfural from sugarcane bagasse



**Scheme 2:** Synthesis of 1,3-bis[(furan-2-yl)methylene]thiourea

1400 °C. UV-Visible spectrophotometer (Searchtech 752 N, USA) was used to obtain absorption maximum ( $\lambda_{\max}$ ) from absorption spectrum of the BFMT dye solution through plot of varied wavelength against corresponding absorbance [2]. Fourier transform infrared (FTIR) spectroscopy was used as described in previous study [2]. BFMT dye was further characterized by nuclear magnetic resonance (NMR) spectroscopy according to Min et al. [19].

#### 2.2.4 Antimicrobial properties of synthesized BFMT dye

Antibiotic activities of BFMT dye using Agar-well diffusion technique, against *Staphylococcus aureus* (Gram-positive), *Pseudomonas aeruginosa* and *Xanthomonas axonopodis* (Gram-negative) bacteria were determined according to ATCC 25,923, 29,212 and 27,853 respectively.

Antifungal activities of BFMT dye were observed using Agar-well diffusion technique, against *Fusarium oxysporum*, *Colletotrichum gloeosporioides* and *Cercospora zeae-maydis* fungi. Each of these fungi was cultured aerobically at 27 °C for 72 h on sterile nutrient agar plate (NAP) containing 3 mL of sterile molten potato dextrose agar (PDA). Each of the pure fungi isolates was inoculated (with the aid of 4 mm cork borer, sterile needle and syringe) at the center of the NAP containing PDA (3 mL) mixed with 0.5 mL of BFMT (0.02 g/mL). 0.5 mL of Mancozeb (standard antifungi) impregnated with PDA (3 mL) was equally prepared as control for fungi culture. All the plates were incubated at 27 °C for 72 h. After 72 h, mycelia growth inhibition was measured and calculated in percentage.

#### 2.2.5 Preparation and standardization of BFMT dye solution

Dye solution was prepared as described by Jabar and Odusote [20] with slight modification. Synthesized BFMT dye (500 mg) was accurately weighed into a liter standard flask and DMSO (5 mL) was added to make a paste. DMSO (20 mL) was added to the paste to form solution, acetic acid (10 mL) was added as additive and made up

to marked point with addition of distilled water. The dye solution was standardized by calibration graph, through a plot of varied concentration (100–500 mg/L) against corresponding absorbance.

#### 2.2.6 Dyeing of wool and cotton fabrics

The fabrics (wool and cotton) were dyed as described in previous work [2]. Cotton fabric (5 g each) was introduced into each of the three pairs of equilibrated cups of Roaches dyeing machine (Model IIMB, UK) containing prepared BFMT dye solutions (100 mL of 500 mg/L) of different pH values (1, 3, 5, 7, 9 and 11) at liquor ratio 1:20. The Dyeing temperature of the machine was raised to 70 °C within 30 min of dyeing and held for the next 30 min at 100 rpm rotating speed of dyeing cups. The procedure was repeated for wool fabric. The BFMT dyed fabrics were removed from dyeing machine at the end of dyeing time and washed with water to eliminate surface adhering dye molecules.

#### 2.2.7 Scanning electron microscopy (SEM) of white and BFMT dyed fabrics

Scanning electron microscope (TESCAN Vega TS 5136LM SEM) was used to determine surface morphology of white and BFMT dyed wool and cotton fabrics as described in previous study [21]. The surfaces of the fabrics were coated with a 10 nm thick layer of gold and machine operated at 20 kV.

#### 2.2.8 Measurement of colour and colour strength

Datacolor (500 USA) was used to generate reflectance (R) and CIELab coordinates ( $L^*$ ,  $a^*$ ,  $b^*$ ) of white and BFMT dyed wool and cotton fabrics automatically under  $D_{65}$  10 light as described by Rather et al. [22]. The colour strength (K/S) of the dyed fabrics was obtained from the Kubelka–Munk equation (Eq. 1), chroma ( $c^*$ ) and hue ( $h^\circ$ ) were obtained from Eqs. 2 and 3 respectively.

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

$$c^* = (a^{*2} + b^{*2})^{\frac{1}{2}} \quad (2)$$

$$h^{\circ} = \tan^{-1} \frac{b^*}{a^*} \quad (3)$$

where  $L^*$  is lightness,  $a^*$  and  $b^*$  are color axis;  $+a^*$  = red,  $+b^*$  = yellow,  $-a^*$  = green,  $-b^*$  = blue color,  $K$  and  $S$  are reflection and scattering coefficients respectively.

### 2.2.9 Fastness properties of BFMT dyed fabrics

Fastness properties of the BFMT dyed wool and cotton fabrics to wash, light and rub were evaluated according to AATCC Test Method 61, 16 and 8 respectively. The fastness property of the dyed fabrics to hot ironing was assessed according to Jabar [23]. The change in color of the test fabric samples was evaluated with the help of gray scale. The numerical ratings (1–8) were assigned for light fastness property [2]; (1–5) were assigned for wash, rub and hot ironing fastness properties [24].

### 2.2.10 Antimicrobial properties of BFMT dyed fabrics

Antibiotic activities of BFMT dyed wool and cotton fabrics were observed using Agar-well diffusion technique, against *S. aureus*, *P. aeruginosa* and *X. axonopodis* bacteria according to AATCC Test Method 147.

Antifungi activities of BFMT dyed wool and cotton fabrics were observed using Agar-well diffusion technique, against *F. oxysporum*, *C. gloeosporioides* and *C. zea-maydis* fungi according to AATCC Test Method 30.

### 2.2.11 Measurement of UV-A and UV-B and determination of UPF Factor of dyed fabrics

UV-Visible spectrophotometer equipped with integrating sphere accessory (Thermo scientific DRA-EV-600–75 Fabric analyzer) was used to measure %Transmission of UV-A, UV-B rays and UPF was determined according to AATCC Test Method 183.

### 2.2.12 Mechanical properties of white and BFMT dyed wool and cotton fabrics

Tensile strength and elongation tests on white and BFMT dyed fabrics were performed on Universal Testing Machine (Instron model 3369) according to the ASTM D 5035, while

stiffness was evaluated on the same machine according to the ASTM D 1388.

### 2.2.13 Air permeability, water vapor permeability and watability properties of white and BFMT dyed wool and cotton fabrics

Air and water vapor permeability of white and BFMT dyed fabrics were measured using test device (Textest Fx Switzerland) according to ASTM D737 and evaporating dish method according to ASTM E96-10 respectively. While watability properties of the fabrics were determined using capillary rise method.

### 2.2.14 Acute toxicity test ( $LD_{50}$ ) of BFMT dye

Ten adult mice (24.5–27.1 g) were paired in 5 different cages, fed for five days with food and water to familiarize them to the environment. 50, 100, 150, 200 and 250 mg/kg (weight of BFMT/ weight of mouse) of BFMT dye thoroughly mixed with 1 mL saline solution were orally administered to the mice (depending on body weight) on day 6 in first to fifth cage respectively. They were strictly observed for death and toxicity signs for 24 h and observation was extended to the next 24 h.

## 3 Results and discussion

### 3.1 Characterization of synthesized BFMT dye

#### 3.1.1 Physicochemical properties

Synthesized BFMT dye of molecular weight 232 was obtained as reddish brown crystal with yield of 70.03% (Eq. 4) and melting point was between 190 and 192 °C. The yield of synthesized BFMT dye is relatively high compared to 18.37% yield obtained from extraction of *B. ferruginea* dye from plant material in previous study [2]. This observation confirmed possibility of synthesizing BFMT dye to industrial scale. High melting point indicated that synthesized dye is thermally stable for most industrial applications.

$$\text{Yield}(\%) = \left( \frac{\text{Experimental yield}}{\text{Theoretical yield}} \right) \times 100\% \quad (4)$$

Elemental analysis: 'Found (%): C, 56.90; H, 3.55; N, 12.00; O, 13.77; S, 13.89'; 'molecular formula  $C_{11}H_8N_2O_2S$ '; required (%): C, 56.88; H, 3.47; N, 12.06; O, 13.78; S, 13.81.

The synthesized BFMT dye was slightly soluble in water and ethanol, insoluble in n-hexane and benzene, soluble in acetone and readily dissolved in DMSO at room

temperature. Therefore, DMSO was a very good solvent for BFMT dye. This implied that BFMT dye solubility increased with increase in solvent polarity.

### 3.1.2 UV-Visible spectrometry

UV-Visible electronic spectrum of BFMT dye solution is displayed in Fig. 1. The three major absorption peaks in the spectrum of BFMT dye solution appeared at 385, 485 and 650 nm, where peak at wavelength (485 nm) correspond to wavelength of maximum absorption ( $\lambda_{\max}$ ). Absorption peaks at 385, 485 and 650 nm can be attributed to the  $\pi \rightarrow \pi^*$  and  $n \rightarrow \pi^*$  transitions of C=C (conjugated bond) and C=S (heteroatom) respectively. Therefore, Peak at

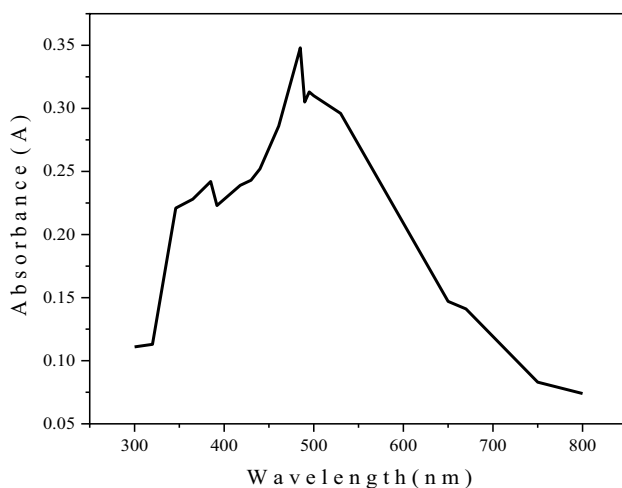


Fig. 1 Absorption spectrum of BFMT dye solution

485 nm is associated with chromophore, which is main color imparting group, peaks at 385 and 650 nm associated with chromogen, which is color parent compound [25]. UV-Visible spectrometry analysis confirmed synthesized BFMT dye to be of reddish brown complementary color with  $\lambda_{\max}$  485 nm.

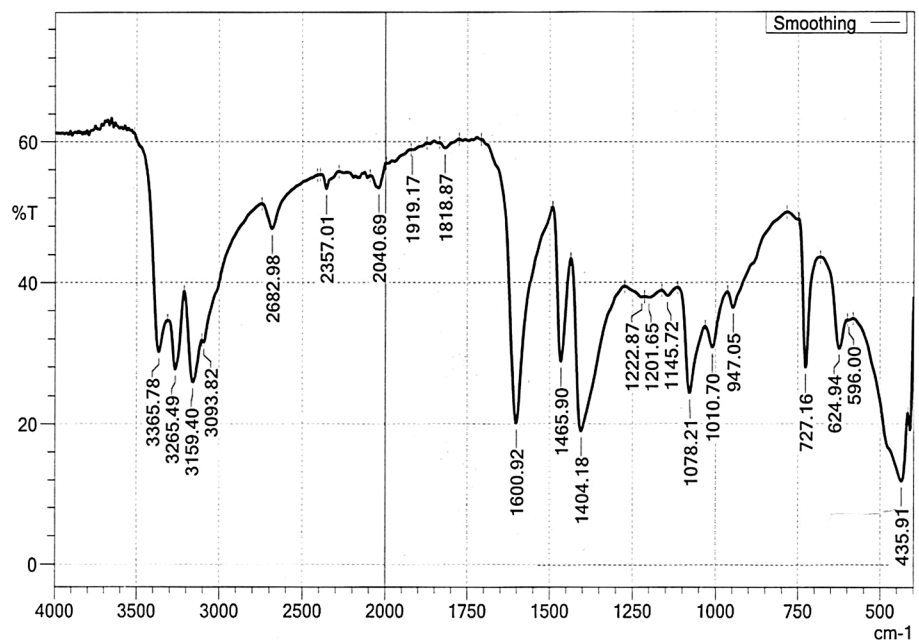
### 3.1.3 FTIR spectrometry

FTIR spectrum of BFMT dye is shown in Fig. 2. Absorption bands that appeared between 3365 and 3265  $\text{cm}^{-1}$  indicated presence of -OH group of the adsorbed water molecule [53]. However, absorption bands in the region 3159–3093  $\text{cm}^{-1}$  correspond to the existence of C=C conjugated bond [26]. The absorption peak at 2682.98  $\text{cm}^{-1}$  is an indication of stretching vibration of -CH group [27]. The sharp peak at 1600.92  $\text{cm}^{-1}$  correspond to presence of C=S heteroatom in BFMT dye. The absorption peak that appeared between 1465.90 and 1404.18  $\text{cm}^{-1}$  confirmed C=N functional group in BFMT dye. The peaks between 1222.87 and 1145.72  $\text{cm}^{-1}$  confirmed -OH functional group of adsorbed moisture and peaks between 1078.21 and 1010.70  $\text{cm}^{-1}$  confirmed C-O stretching vibration [28]. FTIR analysis correlated with proposed structure of synthesized BFMT dye.

### 3.1.4 NMR analysis

$^1\text{H}$  NMR (600 Hz,  $\text{DMSO-d}_6$ ) spectrum shown in Fig. 3 revealed singlet signals at  $\delta$  2.5 and 3.3 ppm indicating proton of DMSO used as solvent and aromatic protons of BFMT dye respectively, while singlet signal at 7.00 ppm

Fig. 2 FTIR spectrum of BFMT dye



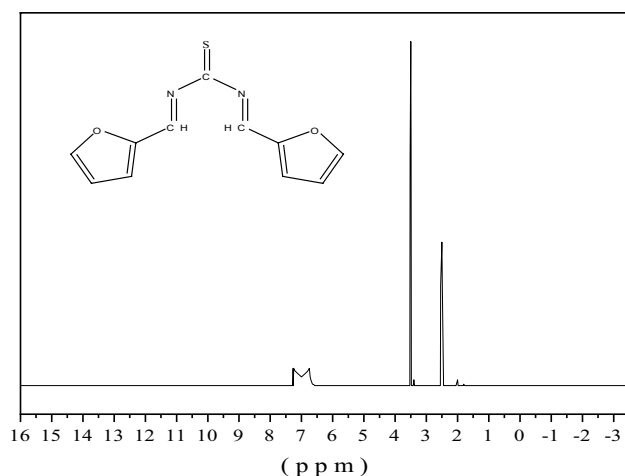


Fig. 3  $^1\text{H}$  NMR spectrum of BFMT dye

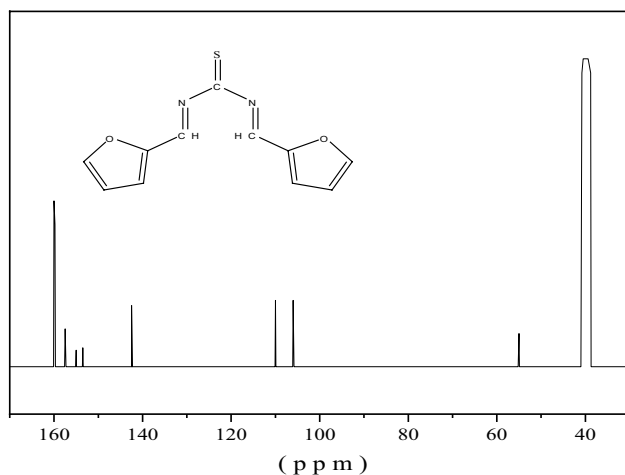


Fig. 4  $^{13}\text{C}$  NMR spectrum of BFMT dye

was characteristic of acrylate proton [19]. Consequently,  $^1\text{H}$  NMR spectrum conformed to proposed structure. Figure 4 shows  $^{13}\text{C}$  NMR (150 Hz,  $\text{DMSO}-d_6$ ) spectrum. Peak at  $\delta$  55 ppm was assigned to carbon atom of DMSO used as solvent. Peaks at  $\delta$  105 and 110 ppm were characteristic of aromatic carbon ( $\text{C}=\text{C}$ ) and acrylate carbon ( $\text{C}=\text{N}$ ) respectively. Peak at  $\delta$  157.5 ppm was ascribed to methoxyl carbon ( $\text{C}-\text{O}$ ), while that at  $\delta$  160 ppm was characteristic of thio carbon ( $\text{C}=\text{S}$ ) [29]. Therefore, NMR spectra (Figs. 3, 4) agreed with proposed structure of synthesized BFMT dye.

### 3.2 Antimicrobial properties

The result of antimicrobial activity of BFMT dye on *S. aureus*, *P. aeruginosa* and *X. axonopodis* is shown in Table 1. The moderate diameter of inhibition zone of BFMT dye as compared to Streptomycin sulphate (standard antibiotic)

Table 1 Diameter of inhibition zones (mm) at 24 h incubation

Sample	Bacteria		
	<i>S. aureus</i>	<i>P. aeruginosa</i>	<i>X. axonopodis</i>
BFMT	4.00	4.00	4.00
STREP	17.00	14.00	20.00

STREP Streptomycin sulphate (Standard antibiotic)

Table 2 Inhibition activity (%) of BFMT on selected fungi at 72 h of incubation

Sample	Fungi		
	<i>F. oxysporum</i>	<i>C. gloeosporioides</i>	<i>C. Zeae-maydis</i>
BFMT	57.00	85.71	60.00
Mancozeb	60.00	90.00	82.00

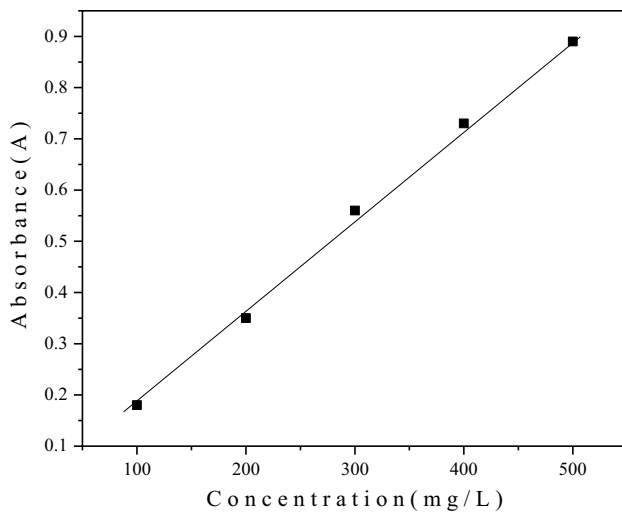
Mancozeb is a commercial antifungal used as a control

on the tested bacteria indicated that BFMT dye could be applied safely in the treatment of infections caused by any of *S. aureus*, *P. aeruginosa* and *X. axonopodis* in its present concentration of 0.2 mg/mL [30]. There might be increase in zone of inhibition, if the concentration of the dye is increased according to Ali and El-Mohamedy [31].

The antifungal activities of BFMT dye on *F. oxysporum*, *C. gloeosporioides* and *C. zeae-maydis* shown in Table 2 varied from very good to excellent when compared to that of Mancozeb (standard antifungi). *C. gloeosporioides* and *F. oxysporum* were highly affected by BFMT dye and it showed very good effect on *C. zeae-maydi*. This indicated that the BFMT dye was a very good antifungal agent against all tested fungi. The presence of colored imparting agent ( $\text{C}=\text{S}$ ) might be responsible for the inhibiting growth and killing of fungi by penetrating into the fungi lipopolysaccharide cell membrane [7]. These observations suggested the synthesized BFMT dye to be a very good agent for treatment of fungal infections.

### 3.3 Dyeing of wool and cotton fabrics with BFMT dye

After dyeing period of 1 h, BFMT dyed wool and cotton fabrics were cleaned, dried and surface morphology assessed by SEM. Fabric dye exhaustion was obtained from interpolated dye effluent concentration (Fig. 5) and presented in Table 3. It can be seen from Table 3 that fabrics dyed in acidic medium have excellent dye exhaustion, but those dyed in basic medium have fair dye exhaustion. As pH of dye solution increased from 1 to 5, quantity of dye absorbed by the fabrics increased. The quantity of dye absorbed by the fabrics decreased as pH value of the dye



**Fig. 5** Calibration curve of BFMT dye in aqueous solution at room temp. and  $\lambda_{\max}$  485 nm

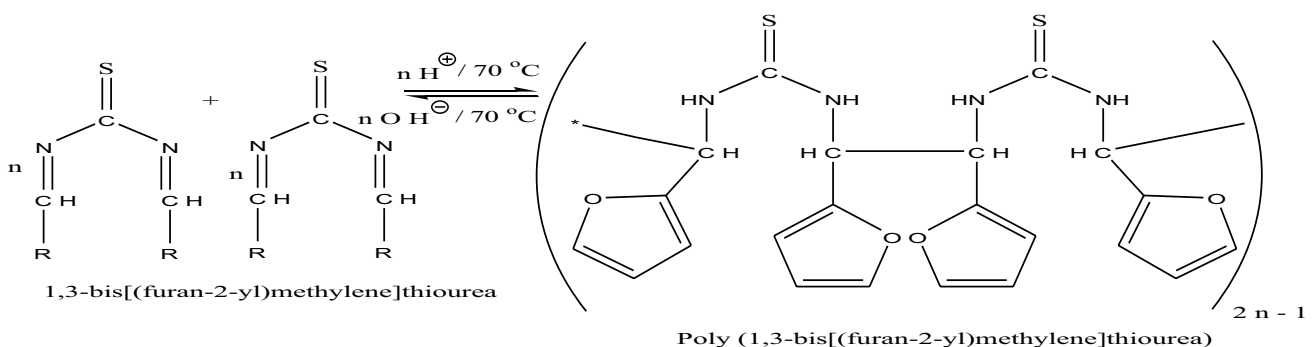
medium increased from 5 to 11. The high dye exhaustion noticed between pH 1 and 5 might be due to migration of BFMT dye molecules from solution onto fabrics matrices as low molecular weight compound, later underwent acid catalysed *in-situ* polymerization reaction as dyeing temperature was raised to 70 °C. The giant dye molecule in the fabrics matrices became bigger to leach out from pore sites on the dyed fabrics (Scheme 3).

Where R = furan.

The lower dye exhaustion at pH 1 and 3 when compared to that of pH 5 might be as a result of competition between hydrogen ion surface charged dye molecules and hydrogen ion from strong acidic medium for hydroxyl ion on the fabrics. Decrease in dye exhaustion noticed as pH value of dye medium increased from 5 to 11 (alkaline region) might be as a result of electrostatic repulsion between hydroxyl ion of alkaline dye medium and hydroxyl ion on fabrics. Equally in the alkaline medium, formation of dye giant molecule did not occur and dye molecules have opportunity of migrating out from fabrics matrices as they were being dyed [25]. Dye molecules migrating in and out of fabrics matrices as they are

**Table 3** Dye exhaustion of BFMT dyed wool and cotton fabrics

Fabric	pH	Effluent Abs. (A)	Effluent Conc. (mg/L)	Qty of dye Absorbed (mg/L)	Dye exhaustion (%)
Dyed wool	1	0.23	126.67	373.33	74.67
Dyed cotton	1	0.27	148.89	351.11	70.22
Dyed wool	3	0.19	104.44	395.56	79.11
Dyed cotton	3	0.22	121.11	378.89	75.78
Dyed wool	5	0.16	87.78	412.22	82.44
Dyed cotton	5	0.18	98.89	401.11	80.22
Dyed wool	7	0.28	154.44	345.56	69.11
Dyed cotton	7	0.31	171.11	328.89	65.78
Dyed wool	9	0.46	254.44	245.56	49.11
Dyed cotton	9	0.50	276.67	223.33	44.67
Dyed wool	11	0.53	293.33	206.67	41.33
Dyed cotton	11	0.54	298.89	201.11	40.22



**Scheme 3:** In-situ polymerization reaction of BFMT dye in dyed fabric

being dyed was more pronounced as pH value increased (Table 3). Therefore, fabrics dyed at pH 5 have optimum exhaustion and were chosen for further analysis on the dyed fabrics.

### 3.4 Morphology of the BFMT dyed fabrics

Figure 6 shows the surface morphologies of wool and cotton fabrics. White cotton and wool fabrics have rough surfaces showing voids and pores that are potential sites for absorption of synthesized BFMT dye onto the fabrics from aqueous solution. They equally showed low molecular orientation, where their molecules were arranged at random, irregular, far apart from one another and fibre longitudinal axis (Figs. 6a, b). Therefore, it can be said that white fabrics were of amorphous structure. This physical property of white fabrics attributed high moisture and dye absorption to them according to Mather and Wardman [32]. After dyeing, surface morphologies of the dyed fabrics were similar to those of white fabrics. This observation indicated that dyeing wool and cotton fabrics with BFMT dye did not affect physical structure of the fabrics and that dyed fabrics were of amorphous structure, just like white fabrics Fig. 7.

### 3.5 Colour characteristics of BFMT dyed fabrics

CIELab coordinates color measurement of white and BFMT dyed fabrics is presented in Table 4. The higher  $L^*$  values of white fabrics showed that they are lighter (more luminous) than their corresponding dyed fabrics according to Bhuiyan et al. [33]. The positive values of  $a^*$  and  $b^*$  of the dyed wool and cotton fabrics indicated appearance of their hue

within red and yellow zone on CIEL color space [34]. The value of  $b^* > a^*$  in BFMT dyed cotton fabric confirmed its appearance color closer to yellow more than red color, justifying its dull reddish brown color. The value of  $b^* < a^*$  in BFMT dyed wool fabric showed that its appearance color is closer to red more than yellow, justifying its bright reddish brown shade. These observations were authenticated by hue angle ( $h^\circ$ ) value of dyed cotton fabric greater than that of dyed wool fabric Fig. 8.

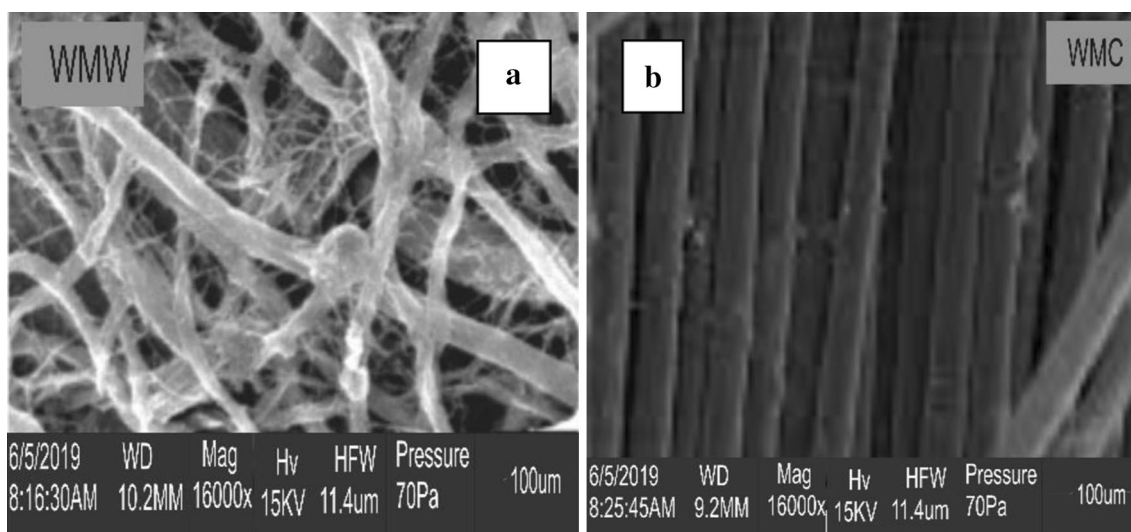
The  $C^*$  values of dyed fabrics showed that brightness of the fabrics was in order dyed wool > dyed cotton.

### 3.6 Color strength of BFMT dyed fabrics

The reflectance (R) and calculated color strength (K/S) values of the dyed fabrics are shown in Table 5. The K/S values of white fabrics are less than their corresponding BFMT dyed fabrics, probably due to deeper color of the dyed fabrics. The higher K/S value of dyed wool compared to that dyed cotton might be due to its brighter hue as earlier observed in their  $C^*$  values. This observation agreed with Rather et al. [22] who stated that the deeper the colour of dyed fabric, the higher the color strength value (K/S).

### 3.7 Fastness properties of BFMT dyed fabrics

BFMT dyed fabrics have fastness properties ranging from very good to excellent (Table 6). The outstanding fastness properties of both dyed wool and cotton fabrics might be as a result of *in-situ* polymerization of BFMT dye in fabric matrix at dyeing temperature of 70 °C (Scheme 3). The giant dye molecules which are bigger than fabric pore sizes conferred excellent (5) fastness properties to wash,



**Fig. 6** Surface morphologies of **a** wool fabric and **b** cotton fabric



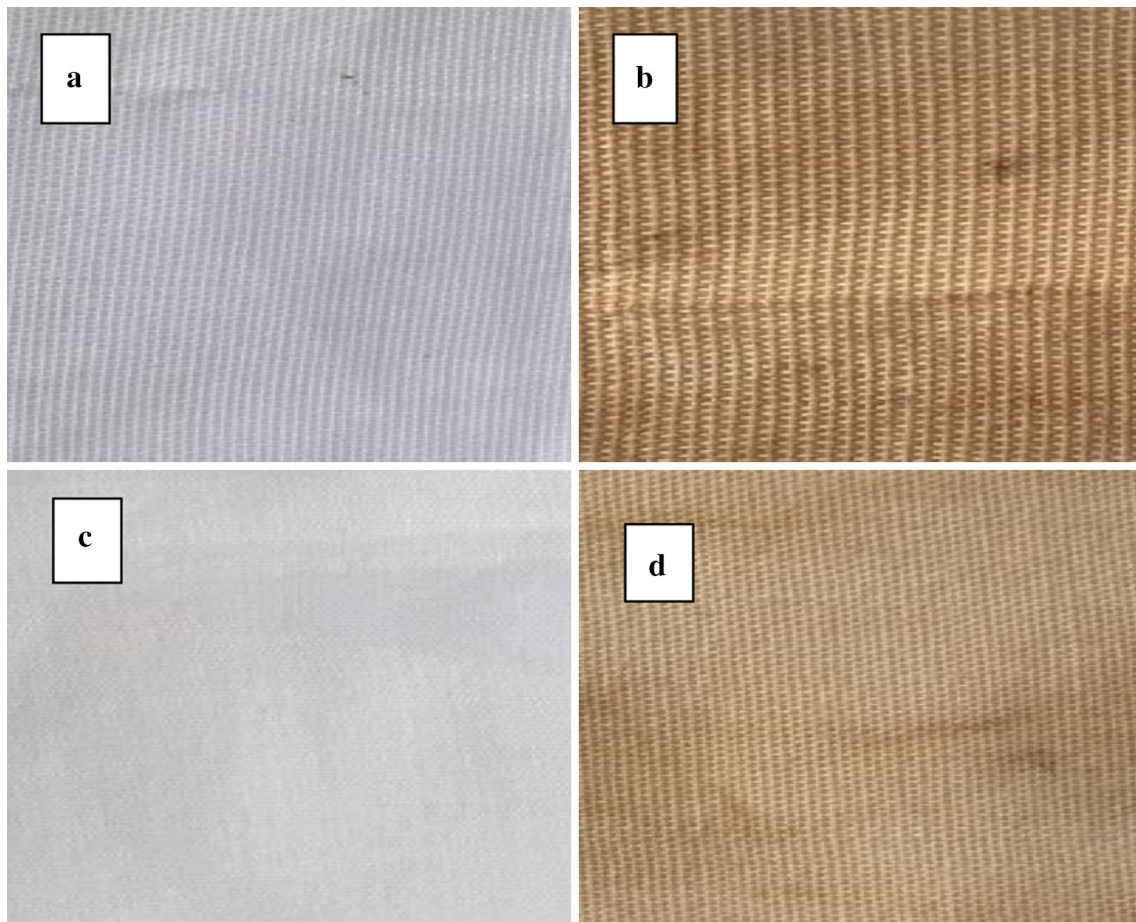


Fig. 7 **a** white wool fabric, **b** BFMT dyed wool fabric, **c** white cotton fabric and **d** BFMT dyed cotton fabric

**Table 4** Color characteristics of white and BFMT dyed fabrics

Fabric	L*	a*	b*	C*	h°
White wool	89.2	-2.3	11.4	11.63	-78.59
BFMT dyed wool	33.9	16.8	16.6	23.62	44.66
White cotton	97.8	-0.3	2.7	2.68	-83.66
BFMT dyed cotton	34.3	16.1	16.9	23.34	46.40

L\* = fabric lightness, +\*a = red, -\*a = green, +\*b = yellow, -\*b = blue, \*c = color chrome and h° = hue angle

rubbing, perspiration and hot ironing on the dyed fabrics. This singular factor might equally be responsible for very good (6) fastness property of dyed fabrics to light.

**3.8 Blocking UV ray and ultraviolet protection factor (UPF) of white and BFMT dyed fabrics**

Figure 9 shows UPF, UV-A and UV-B percentage blocking of white and BFMT dyed fabrics. The wool fabric has inherent high UV ray and UPF protective properties when

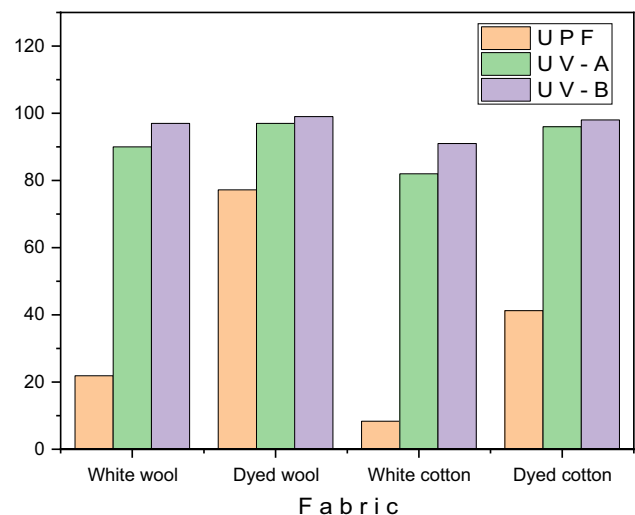


Fig. 8 UV ray and ultraviolet protection factor (UPF) of white and BFMT dyed fabrics

**Table 5** Reflectance and color strength of white and BFMT dyed fabrics

Fabric	R	K/S
White wool	0.65	0.19
BFMT dyed wool	0.15	4.82
White cotton	0.82	0.04
BFMT dyed cotton	0.18	3.74

compared to cotton fabric. White wool fabric has good UPF and its cotton counterpart has poor UPF [35]. Dyeing enhanced UPF property of fabrics as both BFMT dyed wool and cotton fabrics have excellent UPF. This observation agreed with Mather and Wardman [32]. If BFMT dyed fabrics were to be worn under sun light, their absorbed dye molecules are capable of arresting UV ray and get excited to higher electronic energy level (very active radical state). The absorbed BFMT dye would then discharge the absorbed energy in form of radiation or heat and return to ground state with longer wavelength (reflecting bright brown color as complementary color), where by protecting the skin of the wearer from being burnt, premature aging and induced cancer [32].

### 3.9 Antimicrobial properties of BFMT dyed fabrics

The bacterial reduction (*S. aureus*, *P. aeruginosa* and *X. axonopodis*) on white and BFMT dyed fabrics are shown in Table 7. In white fabrics, no antibacterial activity was observed against tested bacteria. However, wool and cotton fabrics dyed with BFMT dye showed a moderate enhancement of antibacterial property against all tested bacteria with moderate mortality rate of bacteria.

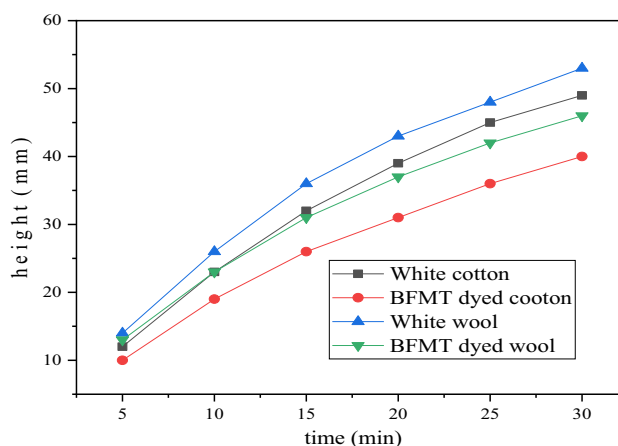
Table 8 shows fungal reduction (*F. oxysporum*, *C. gloeosporioides* and *C. Zeae-maydis*) on white and BFMT dyed fabrics. Just like in antibacterial property of white fabrics, the white fabrics have no antifungal activity. Astonishingly, BFMT dyed wool and cotton fabrics have excellent mortality rate on tested fungi as record showed > 70% fungi destruction in all tested fungi.

Generally, microorganisms are covered by lipopolysaccharides outer cell membrane according to Abu-Dief and Mohamed [10]. If in contact with microorganisms, lipophilic nature of novel BFMT (Schiff base) dyed wool and

**Table 6** Fastness properties of BFMT dyed fabrics

Fabric	Fastness properties							
	Wash			Rub Dry	Perspiration		Hot Ironing	Light
	cc	cs	cw		Alkaline	Acid		
Dyed wool	5	5	5	5	4–5	5	5	6–7
Dyed cotton	5	5	5	5	5	4–5	5	6–7

cc color change; cs color staining of cotton; cw color staining of wool



**Fig. 9** Wetability test on white and BFMT dyed fabrics

**Table 7** Percentage bacterial reduction of white and BFMT dyed fabrics

Fabric	Bacterial reduction (%)		
	<i>S. aureus</i>	<i>P. aeruginosa</i>	<i>X. axonopodis</i>
White wool	–	–	–
Dyed wool	31.23	35.89	23.62
White cotton	–	–	–
Dyed cotton	32.06	36.41	34.2

cotton fabrics has potential ability to penetrate into the lipid outer cell membrane of microorganisms, thus inhibits growth, which might eventually lead to the death of microorganisms according to Sayed et al. [36]. Therefore, world can seize the opportunity of producing medical face masks from BFMT dyed textile materials to reduce community transmission of microorganism born diseases (such as Covid-19) according to Sayed et al. [36].

### 3.10 Mechanical properties of the BFMT dyed fabrics

The results of mechanical properties of the white and BFMT dyed fabrics are presented in Table 9. Dyeing enhanced both tensile strength and stiffness of the wool

**Table 8** Percentage fungal reduction of white and BFMT dyed fabrics

Fabric	Fungal reduction (%)		
	<i>F. oxysporum</i>	<i>C. gloe- osporioides</i>	<i>C. Zeae-maydis</i>
White wool	–	–	–
Dyed wool	93.41	92.17	72.05
White cotton	–	–	–
Dyed cotton	92.36	91.83	71.33

and cotton fabrics, while their elongation was reduced [37]. The mechanical properties of BFMT dyed cotton fabric is more influenced than that of BFMT dyed wool fabric. Better crystalline structure of BFMT dyed cotton fabric than wool counterpart might be responsible for higher mechanical properties. This observation correlated with that of scanning electron microscopic analysis of wool and cotton fabrics.

**3.11 Air permeability, water vapor permeability and watability properties of white and BFMT dyed wool and cotton fabrics**

Table 10 shows values of air and water vapor permeability of white and BFMT dyed fabrics. The higher air and water vapor permeability of wool fabric can be attributed to easier passage of air and water vapor through wool fabric due to higher spacing among the yarns inside the wool fabric cross sectional area, when compared to cotton fabric. This observation validated surface morphological images of wool and cotton fabric in Fig. 6. An insignificant change observed in values air and water vapor of the fabrics after dyeing was an indication that the rate at which air and water vapor diffused through white and BFMT dyed fabrics was virtually equal. Therefore, dyed fabrics will provide thermal comfort, good air and sweat absorption as well as good air and sweat release property to the consumers just like white fabrics.

Figure 9 displays watability property of wool and cotton fabrics. The quantity of water absorbed by the fabrics

**Table 9** Tensile strength, elongation and stiffness of white and BFMT dyed fabrics

Fabric	Mechanical properties			
	Tensile strength	Elongation	Stiffness	Stiffness
	(kgm/cm)	(%)	in weft (mg)	in warp (mg)
White wool	44.8	45.7	405.7	608.3
BFMT dyed wool	47.5	43.8	412.2	643.5
White cotton	56.4	31.9	461.6	703.6
BFMT dyed cotton	60.1	28.7	473.1	716.4

**Table 10** Air and water vapor permeability of white and BFMT dyed fabrics

Fabric	AP (cm <sup>3</sup> cm <sup>-2</sup> s <sup>-1</sup> )	M (g)	A × 10 <sup>-4</sup> (m <sup>2</sup> )	t (h)	WVP (g m <sup>-2</sup> h <sup>-1</sup> )
White wool	508.83	0.56	9.07	20	740.90
BFMT dyed wool	502.48	0.55	9.07	20	727.67
White cotton	483.61	0.54	9.07	20	714.44
BFMT dyed cotton	475.04	0.52	9.07	20	687.98

Where; AP is air permeability, M is loss in mass of fabric and water containing evaporating dish assembly over time t, A is area of exposed fabric sample, t is time between successive of assembly and WVP is water vapor permeability

due to capillary forces was time dependent (Fig. 9). It was noticed that as absorption time increased, the rate of water absorption reduced due to reduction in available pore sites of the fabrics. The inconsequential difference in water absorption pattern between white fabrics and BFMT dye fabrics confirmed their hydrophilic nature to be nearly equal. Therefore, water absorbency properties of both white and dyed fabrics will protect consumers’ skin from sudden change in weather conditions (act as heat reservoirs), occurrence of static electric discharge and enhance removal of water burn stain in the fabrics.

**3.12 Acute toxicity test (LD<sub>50</sub>) of BFMT dye**

Neither death nor toxicity sign was seen in mice inside cages 1–3 orally administered with 50–150 mg/kg of BFMT dye respectively within 48 h of observation (Table 11). Mice in cage 4 showed neither death nor toxicity sign within 24 h they were orally administered with BFMT dye, but developed weakness and drowsiness within next 24 h. None of the mice in the fifth cage died, but they showed signs of weakness and drowsiness within 24 h they were orally administered with BFMT dye (250 mg/kg). Hence, BFMT dye cannot be classified as highly toxic substance since it did not cause harm to any of the mice administered with dose between 50 and 150 mg/kg according to Loomis and Hayes [38] who stated that any substance that cause

**Table 11** Acute toxicity ( $LD_{50}$ ) test on BFMT dyed

Parameter	Time (h)	S/N	Cage containing BFMT as oral dose				
			1	2	3	4	5
			BFMT (mg/kg)				
No of death in mice	24		50	100	150	200	250
	48		0	0	0	0	0
Physical change Observed	24		Nil	Nil	Nil	Nil	WD
	48		Nil	Nil	WD	WD	WD

*Nil* nothing, *WD* weakness and drowsiness

harm to animal orally administered with dose below 5 mg/kg could be regarded as highly toxic substance.

## 4 Conclusion

Novel BFMT dye was successfully synthesized from condensation reaction between sugarcane bagasse derived furfural and thiourea.

Multifunctional dyed fabrics obtained from application of BFMT dye on wool and cotton fabrics have very high and moderate inhibition rate on tested fungi and bacteria respectively. Therefore, the dyed fabrics can be utilized in production of medical face masks for preventing microorganisms born diseases.

Excellent UPF of the BFMT dyed fabrics indicated their potential ability as sun screen wears, which may protect the consumers from harmful effect of sun burn.

Excellent exhaustion and fastness properties of the dyed fabrics are indicators of prolong usage of the fabrics without fading out color on them.

High mechanical properties of the dyed fabrics implied that they are potential materials for production of engineering laboratory coats.

Wool and cotton fabrics will provide thermal comfort, good air and sweat absorption as well as good air and sweat release property to the consumers.

## Compliance with ethical standards

**Conflict of interest** The authors declare that they have no conflict of interest.

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