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Characterization of New Coloured Materials on Different Fabrics and Application on Wool Fabrics to Comfort and Ultraviolet Protective Garment

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FABRICS treated with many chemicals and different technologies to give them good functional properties. In this study, new coloured materials were prepared and used for colouring and finishing fabrics in one step. Pyrimidine derivatives are compounds that have attracted much attention in recent years because of their broad spectrum of biological activities. We have synthesized various pyrimidine derivatives such as aminothioxopyrido (2,3-d) pyrimidine derivatives as coloured materials, which are used in dyeing different textile materials. The effect of the nature of various substituent on this type of pyrimidine derivatives as dye (new coloured materials) behavior has been studied on various types of textiles; all synthesized compounds were examined for their dyeing properties on different fabrics such as wool, acrylic, viscose and polyester fabrics. The chemical structures of the synthesized compound 1-3 have been elucidated by spectroscopic data analysis and are in good agreement with the proposed structures. Moreover, the colour strength was examined in detail. The colour strength of dyed textiles and the fastness of the compounds were determined. Wool fabrics got better dyeability than other fabrics. The effect of pH dyeing bath, dyeing temperature and dye concentration for the new coloured (amino-thioxopyrido (2,3-d) -pyrimidine derivatives) were studied.

In addition, the effects of the properties of the new coloured materials on the performance of woolen garments were identified and some properties of coloured fabrics such as weight, thickness, roughness, air permeability, thermal insulation and bending modulus examined. The moisture transport property was measured by a wicking test in the vertical and horizontal directions. The treated fabric exhibited excellent ultraviolet protection without significant discrepancy in its inherent physico-mechanical properties. The effect of new dyes on the performance characteristics of woolen garments was evaluated using a radar chart.

Keywords: Textile, Pyrimidine derivatives, Dyeing, Fastness, UPF, Functional properties, Wool garment performance.

Introduction

Heterocyclic compounds containing pyrimidine derivatives and pyridazine are still giving much attention because the interesting pharmacological activities are greatly increasing and textile. [1] These compounds have received considerable attention in recent years because of their broad spectrum of biological activities and pharmaceutical applications. Pyrimidine compounds with different pharmacological

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properties have cyclic systems, a number of articles have been described in the literature. [2-5]. Continuing our work, the synthesis of certain thioxopyrido (2,3-d) -pyrimidine derivatives and (pyrano-2,3-d) -pyridazine derivatives for various fibers has been investigated. [6, 7] .

The versatility of the barbituric acid or thiobarbituric acid moiety is attributed to its ability to serve alternatively as a donor or as a substituent. Mercocyanine dyes that contain barbituric acid as M-substituent are well established and contain barbituric acid or its N-substituent derivatives. [8] Dyeing can be explained in three steps: I) Adsorbed on the fiber surface. II) penetration of the fibers and III) covering of the dye sites in the fiber. [9, 10] Abdallah et al presented that although novel benzimidazole derivatives has been heterocycles of anticancer activity and hydrazono derivatives, it can be used as a dyeing material on different fabrics. [11].

Wool fabrics are regarded as the most valuable fabrics in the garment industry. The dye ability of coarse wool with acid dyes has been improved by bio-carbonization treatments. [12] The treatment with pentaerythritol led to enhancement dyeability with acid dyes on performance of wool garment as well as anti-pilling. [13] Wool fibers pretreated with nano chitosan and natural dye as well as different solvents led to improved the dyeability of wool and wool /acrylic fabrics after. [14, 15]. The thiazolidine derivative was prepared and used as a novel colourant on viscose fabrics treated with nano-kaolin. [16].

In order to improve some functional properties and comfortability for fabrics, we need treatments at various stages, which can be done before or after dyeing. Moreover, as a result of changing the climate, it became necessary for fabrics to offer sunlight protective which known on the term of the UPF (Ultraviolet Protection Factor). Several articles defined that a minimum rating of fabric protection has to exceed 15 UPF as a meaning of good. [17].

In this study, the desired of functional properties were obtained with dyeing in one step. The present work is to investigate the new materials as dyed or coloured materials on different fabrics. This study demonstrates the influence of new coloured materials (pyrimidine derivatives or pyridazine derivatives) on the dyeability and fastness properties of different fibers. The investigation revealed better dyeing conditions such as sutible pH of the dyebath, better dyeing temperature and sutible colour percent of the dye shade . Furthermore, the colour strength (k/s) of the dyed textiles is measured. The results will certainly help to determine the design criteria for clothing to produce high quality coloured clothing.

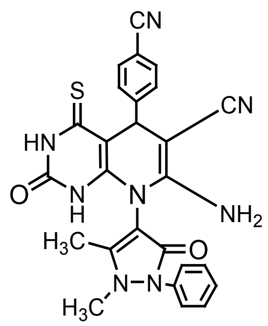
Experimental

Materials

New coloured materials Synthesis

Synthesis of amino-5-(4-cyanophenyl)-1,2,3,4,5,8-hexahydro-8-(2,3-dihydro-1,5-dimethyl-3-oxo-2-phenyl-1H-pyrazolyl)-2-oxo-4-thioxopyrido[2,3-d]pyrimidine-6-carbonitrile derivative (F1):

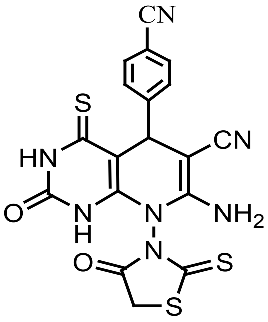
A mixture of 2-Thioxodihydro-4,6(1H,5H)-pyrimidinedione (0.01 mol), 2-(4- cyano-benzylidene) malononitrile (0.01 mol) and (4-aminoantipyrine) (0.01 mol) in acetone (10 ml) in the presence of nano-SPIA (5 mol%) and DMF –DMA (1mL) which was refluxed for 2h at 100 ^{0}C with stirring , then left to cool, filtered off, the solvent was removed under vacuum. The solid that formed was recrystallized from absolute ethanol, dried under vacuum to form the product compound 1.



F1

Synthesis of amino-5-(4-cyanophenyl)-1,2,3,4,5,8-hexahydro-2-oxo-8-(4-oxo-2-thioxothiazolidinyl)-4-thioxopyrido[2,3-d] pyrimidine-6-carbonitrile derivative (F2):

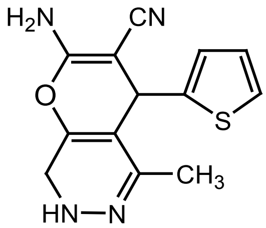
A mixture of 2-thioxo-dihydropyrimidine-4,6 (1H,5H) dione (0.01 mol), 2-(4- nitro-benzylidene) malononitrile (0.01 mol) and (3-aminorochdanine) (0.01 mol) in ethanol (20 ml) in the presence of nano-SPIA (5 mol%) and DMF –DMA (1mL) which was refluxed for 90 min with stirring, then left to cool at room temperature, filtered off , the solvent was removed under vacuum. The solid that formed was recrystallized from absolute ethanol, dried under vacuum to afford compound 2.



F2

Synthesis of amino-7,8-dihydro-5-methyl-4-(thiophenyl)-4H-pyrano[2,3-d]pyridazine-3-carbonitrile derivative (F3):

A mixture of hydrazine hydrate (10 mmol),ethyl 3-oxobutanoate (10 mmol), malononitrile (10 mmol) and 5-hydroxyl-thiophene-2-carboxaldehyde derivative and (10 mmol) was added in ethanol (15 ml) and then nano-SPIA (5 mol%) was added in the presence of DMF-DMA (1 ml). Then the solution was heated under reflux with stirring at 100°C and maintained at this temperature for 3h. After completion of the reaction monitored by TLC, the reaction mixture was left to cool at room temperature and poured with ethanol several times, filtered, dried under vacuum and recrystallized of the precipitate product from acetone to produce compound 3.



F3

Amino-5-(4-cyanophenyl)-1,2,3,4,5,8-hexahydro-8-(2,3-dihydro-1,5-dimethyl-3-oxo-2-phenyl-1H-pyrazolyl)-2-oxo-4-thioxopyrido[2,3-d]pyrimidine-6-carbonitrile derivative (F1) :

Yield: 85%. m.p. 190–192 ^{0}C. ^{1}H-NMR: (500 MHz, DMSO): 1.30 (3H, s, CH_3), 2.25 (3H, s, CH_3), 4.0 (1H, s, CH), 4.3 (1H, s, CH) 6.20 (1H, br s, NH_2), 6.30–6.60 (4H, m, Ar-H aromatic proton), 6.80–7.20 (5H, m, Ar-H aromatic proton), 8.10 (1H, br s, NH), 8.4(1H, br s, NH),

^{13}C NMR(DMSO-d6): 39.50 (CH_3), 39.80 (CH_3), 110.90, 112.80, 116.90, 119.80, 122.90, 127.00, 130.00, 134.10, 135.80, 138.8, 165.90, 169.40 (2CN), 174.60 (C=O), 178.90(C=O), 210 (C=S). IR (KBr): 3200, 3230 (2NH), (1680), 1695 (2C=O), 1640, 1648 (2CN), 1225 (C=S) cm^{1}.

Amino-5-(4-cyanophenyl)-1,2,3,4,5,8-hexahydro-2-oxo-8-(4-oxo-2-thioxothiazolidinyl)-4-thioxopyrido[2,3-d] pyrimidine-6-carbonitrile derivative (F2) :

Yield: 88%. m.p. 240-242^{0}C. ^{1}H-NMR: (500 MHz, DMSO): 1.90 (3H, s, CH_3), 3.9 (2H, s, CH_2), 4.2 (1H, s, CH), 4.3 (1H, s, CH), 6.0 (1H, br s, NH_2), 6.20–6.50 (4H, m, Ar-H aromatic proton), 8.50 (1H, br s, NH), 8.90 (1H, br s, NH), ^{13}C NMR(DMSO-d6): 111.90, 115.80, 116.90, 119.80, 120.90, 127.00, 130.00, 134.90, 138.80, 138.8, 165.40 (2CN), 170.5, 174.60 , (2C=O) , 205-210 (C=S). IR (KBr): 3230, 3250 (2NH), 3180 cm^{-1} (NH_2), 1690-1720 (2C=O), 1640-1645 (2CN), 1220-1225 (2C=S) cm^{-1}.

Amino-7,8-dihydro-5-methyl-4-(thiophenyl)-4H-pyrano[2,3-d]pyridazine-3-carbonitrile derivative (F3):

Yield: 77%. m.p. 180–182 ^{0}C. ^{1}H-NMR: (500 MHz, DMSO): 1.80 (3H, s, CH_3), 3.9 (1H, s, CH), 4.20 (1H, s, CH), 4.40 (1H, s, CH), 6.50 (1H, br s, NH_2), 6.70–6.80 (4H, m, Ar-H aromatic proton), 8.98 (1H, br s, NH), ^{13}C NMR(DMSO-d6): 35.80 (CH3), 111.90, 115.80, 116.90, 118.50, 125.80, 128.00, 130.00, 166.50 (CN). IR (KBr): 3260 (NH), 3200 (NH_2), 1648 (CN), 1690 (CO) cm^{1}.

Fabrics

Acrylic and polyester fabric were supplied by Masr El Mahalla Company, El Mahalla Elkobra, Egypt. A plain woven acrylic fabric weighing is 175 g/m^2 and a plain woven polyester fabric weighing is 155 g/m^2. The both fabrics were washed in 2g/l nonionic detergent (Egyptol) solution at 45 °C for 30 minutes, thoroughly rinsed with cold water and air dried at room temperature. Polyamide (Naylon)knitted fabric from El-Shorbagy Co., Cairo, Egypt. The fibre density is 1.4. g/cm^3, wet tenacity 38 cN/Tex, and dry strength is 44 CN/Tex. The fabrics were washed in 2 g/l nonionic detergent (Egyptol) solution at 45 °C for 30 minutes, thoroughly rinsed with cold water and air dried at room temperature.

Wool fabrics were supplied by Misr for Spinning and Weaving Co., El Mehalla El Kobra, Egypt, and Plan weaved (25 yarns/cm in both weft and warp directions)with twill 2/2 woven structure.Wool fabrics were scoured for 30 min at 30 °C in a 2% nonionic detergent (based on weight of fabric), fabric: liquor ratio, 1: 50. The scoured fabrics were then rinsed with warm water and air dried.

Plain viscose fabric was supplied by Abou El- Ola for Spinning and Weaving Co., 10th of Ramadan City, Egypt. Its weight is 110 g/m^2, number of warps is 375/10 cm and number of weft is 320/10 cm.

Dyeing method

The dyebath was prepared by accurately weighing the newly dyed materials (new coloured materials) to give the prescribed shade (0.5, 1, 2 and 4% w / v). The paste was then dissolved by adding hot boiling water. The coloured solution was adjusted to a different pH of 1 to 7 with acetic acid. The dyed bath was heated to various temperatures at 30, 40, 60, 80 ° C and the sample (acrylic or polyester or polyamide and viscous or wool) was added for various times in a liquor ratio of 1:50. The dyed sample was washed thoroughly in warm then tap water and air dried.

Measurements

1- **Thickness** (mm) was measured according (ASTM-D1777).
2- **Weight** (Gm/m²) was measured according (ASTM-D3776).
3- **Air Permeability** (Cm²/Cm³/S) was measured according to (ASTM-D737).
4- **UPF** was measured according to (ASTM-D6603)
5- **Bending Stiffness** (Kg/Cm2) was measured according to (ASTM-D1388)
6- **Vertical Wicking (**M/S) was measured according to (AATCC–D197)
7- **Horizontal Wicking** (Mm²/S) was measured according to (AATCC–D198)
8- **Thermal Insulation** (MK.m2.w-1) was measured according to (ISO–11092)
9- **Roughness** (µm) surface roughness (SR) suefacoder1700a.
10- **Colour exhaustion %:** using a spectrophotometer. [18]
11- **Washing fastness**: The colour fastness to washing was determined according to the AATCC test method (AATCC Technical Manual, Method 36, (1972), 68, 23, (1993)) using Launder Ometer. [19]

Results and Discussion

Colour strength and washing fastness

Table 1 shows the colour strength of the coloured various fabrics such as wool, acrylic, polyester, nylon and viscose with new coloured materials . The results show that only the woolen fabrics for all three dyed materials (F1, F2 and F3) were dyed with a high yellow shade. It was found that F2 gave the highest colour strength value as F1 and F3. In addition, acrylics take on a light shade compared to all fabrics.

The results of washing fastness of dyed fabrics with the new coloured material (F2) were tabled in Table 2. The data show a slight improvement in alteration and ant- staining for both wool and acrylic fabrics compared to other fabrics. This may be attributed to found a link between the new coloured materials and wool fabrics.

Table 3 illustrates the effect of dyeing bath solution pH, the effect of dyeing temperatures, and the effect of shade percentage on the colour strength of dyed wool fabrics with the new dyed material (the new coloured) (F2). It has been found that the pH of the solution of the new coloured F2 affects the colour strength value. The best pH for acid medium 2-3 gives the highest colour intensity than other pH values. This can be attributed to the properties of the new coloured material. The dyebath also influenced the dyestuff values for dyed wool fabrics, increasing the dyeing temperature increased the tinting strength and the best temperature in the range from 85 °C to 90 °C. Similarly, the dyeing process was dependent on the concentrations of the dyes (F2), which was the percentage hue of the dyes. The results of Table 3 illustrate that the woolen fabrics are coloured yellow, then dark yellow and brown obtained.

The effect of dyeing characteristics of sample properties

Weight

Figure 1 shows that the wool fabrics dyed with F2 has the highest weight (261.6 g / cm2) among the other coloured fabrics and the undyed fabrics has the lowest weight (242.4 g / cm2). The rationale was related to the effect of the new coloured material property on the shrinkage of the wool fabrics, which increases the yarns and picks per unit area and then in units of weight.

TABLE 1. Color strength of colored fabrics with new coloured material.

Kind of Fabrics	Colour strength (K/S value)		
	F1	F2	F3
Wool	12.2	15.62	11.0
Acrylic	3.1	3.4	3.5
Nylon	1.0	1.9	2.5
Polyester	0.3	0.4	0.8
Viscous	0.8	0.9	1.2

Condition of treatments: 1 % (w.o.f) new coloured materials at L: R, 1: 50, at 85°C. pH 5, for 1hr.,

TABLE 2. Washing fastness of dyed different fabrics with F2.

Samples	Washing fastness						
	Alt	St_A	St_C	St_P	St_N	St_V	St_W
Viscose	3	4	4	4-5	4-5	4-5	4
Wool	4-5	4-5	4	4	4-5	4-5	4
Acrylic	3-4	4-5	4	4-5	4-5	4-5	4
Polyester	3	5	4-5	4-5	4-5	4-5	4
Polyamide	3	4-5	4-5	4-5	4-5	4-5	4

Alt.: Colour alteration, St_A: Staining of acrylic fabrics, Stc: Staining of cotton fabrics, St_P: Staining of polyester fabrics, St_N: Staining of nylon fabrics and St_W: Staining of wool fabrics

TABLE 3. color strength of wool fabrics, dyed with F2.

Wool Fabric with F2					
pH *	K/S	Dyeing Temp.**	K/S	Shade %***	K/S
2.0	18.9	70 °C	13.3	0.5	7.77
3.0	12.7	80 °C	15.62	1.0	15.12
4.0	5.9	85 °C	18.9	1.5	18.9
5.0	4.8	90 °C	19.9	2.0	29.18
6.0	3.7	95 °C	21.15	2.5	29.1
7.0	2.2	99 °C	21.05	3.0	29.0

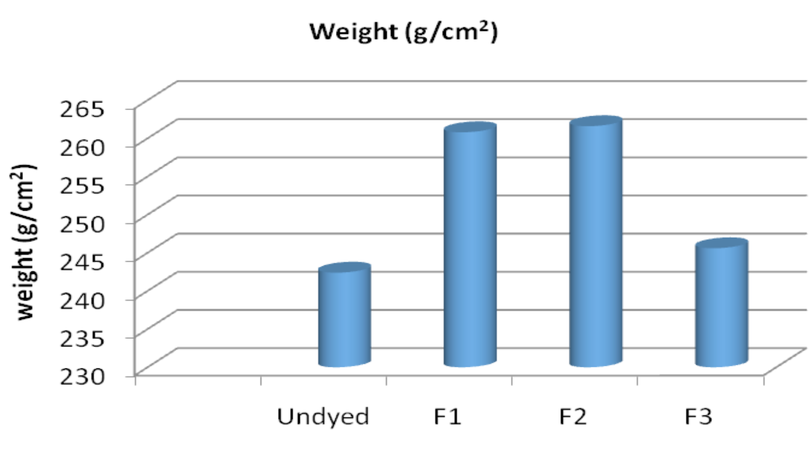


Fig. 1. Weight of coloured wool fabrics.

Thickness

Figure 2 shows that the dyed wool fabrics with both F1 and F2 made the higher thickness (0.87 mm) and (0.88 mm) sequentially compared to other two samples. The explanation was that the swimmer's float was due to the reduction in the distance between yarns and picks due to the shrinkage of the samples. In addition, the result showed that the dyed wool fabrics with F3 is best suited for uncoloured one.

Roughness

The results show a significant effect of the colour material on the roughness properties of the dyed wool fabrics, where undyed fabric shows the large roughness among other samples (39.09 µm), while the dyed with both F1 and F3 new coloured are the lowest (more preferred) as shown in Figure 3. In addition, the results are on the order of magnitude. The effect of dyeing with the new coloured F1 though recognizing a high thickness (about the new coloured F2), achieved the lower roughness.

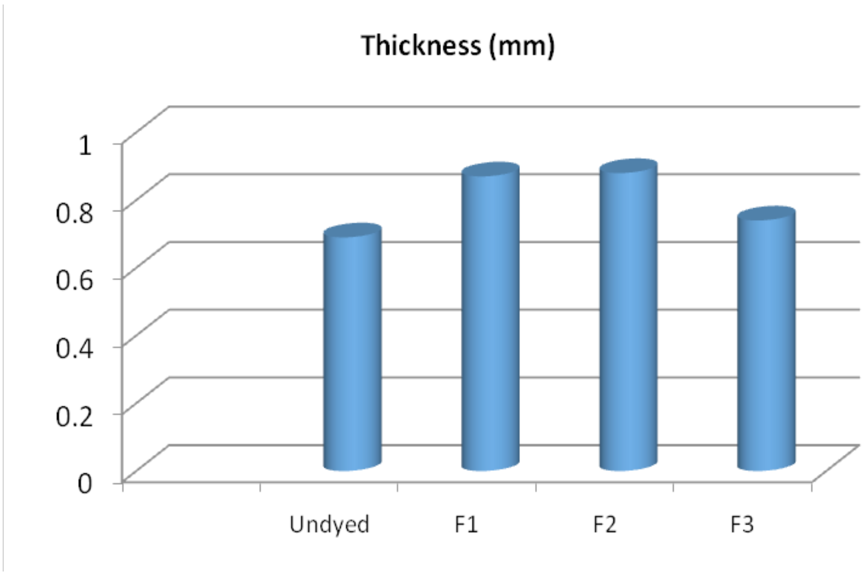


Fig. 2. Thickness of coloured wool fabrics.

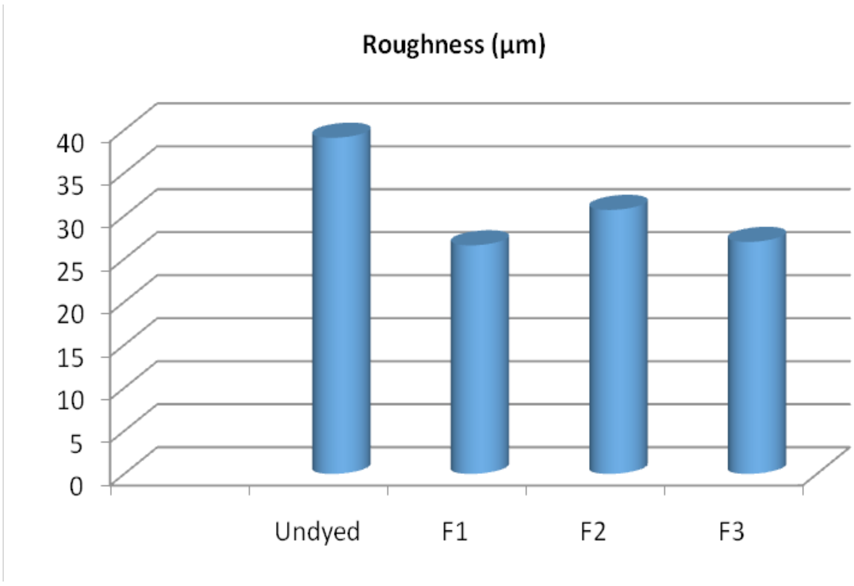


Fig. 3. Roughness of coloured wool fabrics.

Air permeability

The results in Figure 4 show that undyed substances have the highest air permeability, while the dyed wool fabrics with F3 is superior to two other coloured F1 and F2. The interpretation returns to a difference in pore size between yarns and picks within a woven structure due to the new effectiveness of the coloured material properties (which can be deduced from the weight variations of the samples).

Bending modulus

Figure 5 illustrates the strong influence of new dyes on the flexural modulus of woolen fabrics, where undyed fabrics have a higher flexural modulus (290.14 kg/cm^2) than other coloured fabrics. In addition, the result indicated a contradictory situation in which dyed fabrics, despite their high thickness and weight, achieved lower bending modulus.

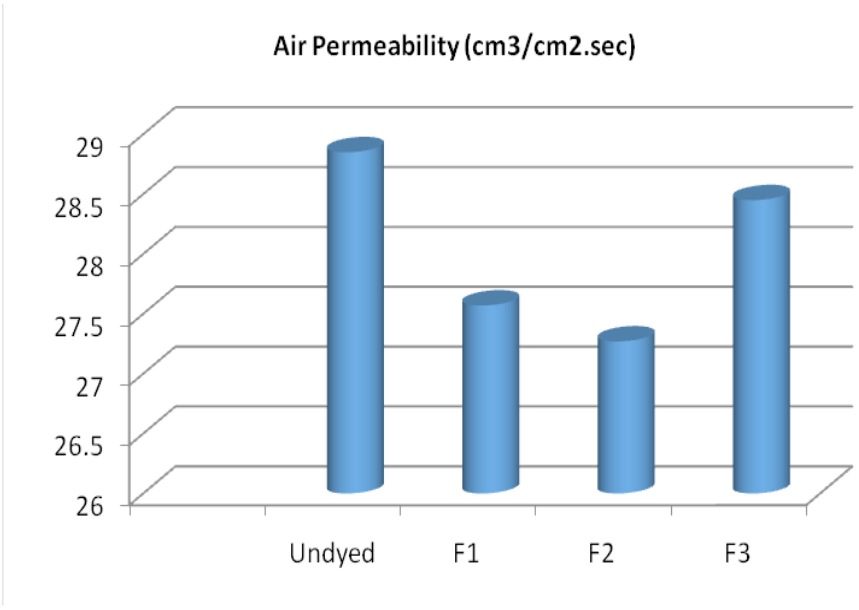


Fig. 4. Air Permeability of coloured wool fabrics.

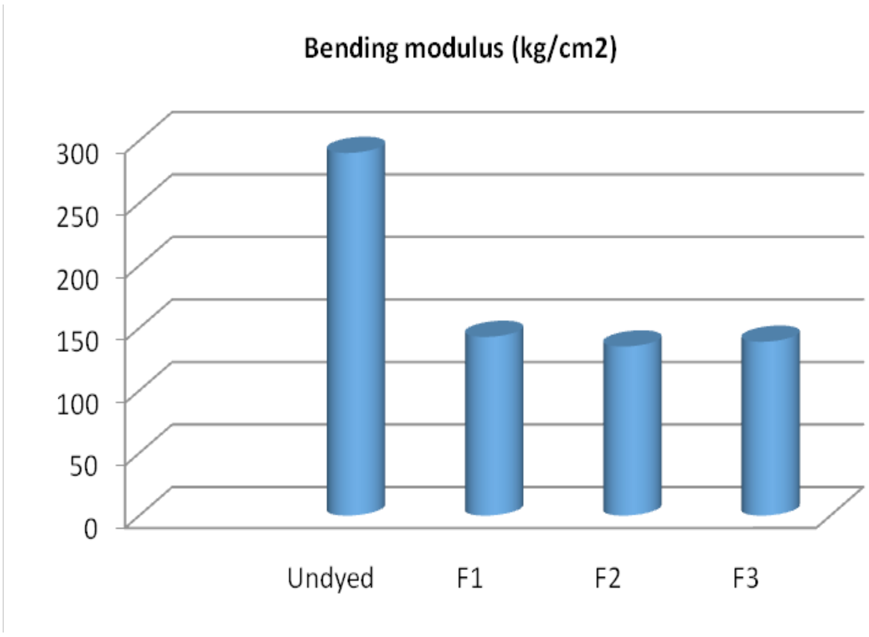


Fig. 5. Bending modulus of coloured wool fabrics.

Thermal Insulation

The results in Figure 6 shows that dyed wool fabrics with F1 and F2 new dyed materials had the highest thermal insulation, followed by dyed wool with F3, while undyed wool fabrics had the lowest. The explanation correlated with the tight interlacing between yarns and picks as a reason for the shrinkage of the fabrics, thus affecting the minimization of pore size in the woven structure.

Samples rating

To determine the best sample in terms of performance of the garment, the radar map area was calculated as shown in Figure 7. The results clarify that the dyed wool fabrics with F2-achieved the highest performance rating than both F1 and F3-dyed wool fabrics. On the other hand, the results showed that the performance of woolen garments increased with new coloured materials and not with undyed patterns that had the least radar surface.

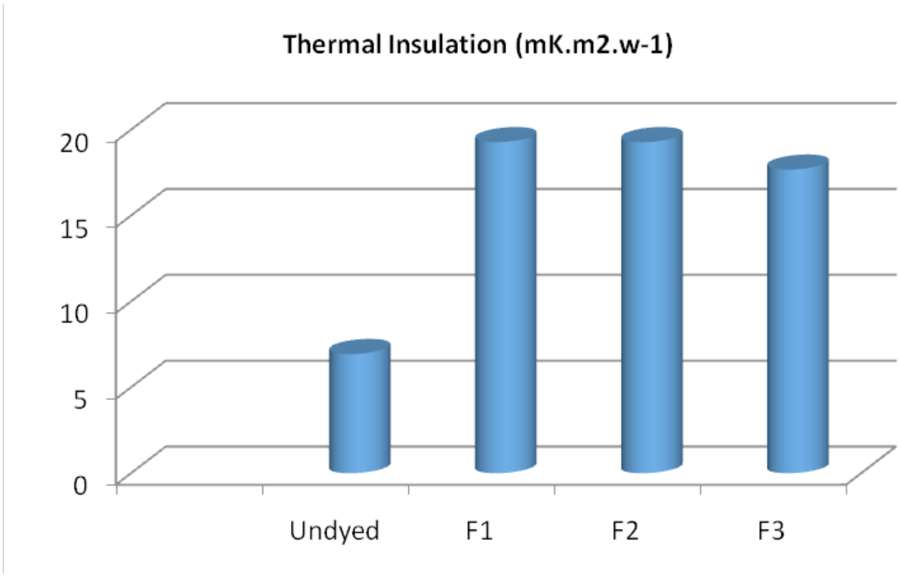


Fig. 6. Thermal Insulation of coloured wool fabrics.

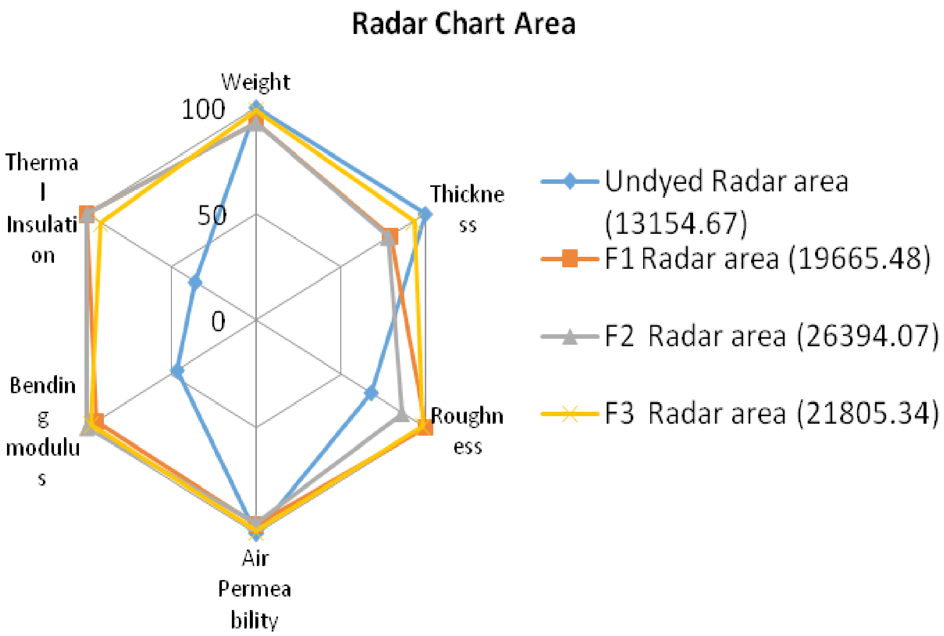


Fig. 7. Sample rating of colored wool fabrics.

Ultraviolet protection

While UPF ultraviolet protection is one of the main features that reflects the ability of garments to protect the human body, especially in Middle Eastern countries (sunny weather). Table 4 shows the UPF rating for wool patterns coloured with new materials. The results show that dyed wool fabrics with new coloured materials give a significant increase UV protection compared to an undyed one. The results indicate that the dyeing with F2 coloured material has very excellent protection 50+ which the highest value compared with the other coloured wool fabrics as well as the uncoloured sample. Followed by dyed wool fabrics with F3 excellent protection 40+. The resistance of the dyed fabrics to ultraviolet radiation indicates that new coloured material (F2) acts as a photoprotective substance as it improves fabric absorbance (more than 98.0%) to UV radiation having wavelengths in the range of 290 to 400 nm (UVA and UVB).

Horizontal & Vertical Wicking Test

To determine the influence of new coloured materials on the moisture-transporting property of woolen fabrics, a wicking test was conducted in the vertical and horizontal directions as shown in Table 5 and Table 6. The results show that the dyed wool fabrics with F3 showed the highest horizontal wicking among other samples, while the dyed wool fabrics with F1 new material realized the second position. Similarly, the result was indicative of the size effect of new coloured materials on the vertical wicking test, in which coloured samples in the warp or weft direction scored higher than uncoloured samples, indicating the possibility of new coloured materials to improve moisture transportation in a horizontal and vertical direction of the wool samples.

TABLE 4. UV protection properties of dyed wool fabrics with new colour materials.

UV Protection Category	UPF Rating	Sample
Very Good protection	30+	Untreated
Very Good protection	35+	F1
Very Excellent protection (ultimate protection)	50+	F2
Excellent protection	40+	F3

TABLE 5. Horizontal Wicking Test.

W_h	(T) Time in second (Sec)	(D2) Distance in the longitudinal direction (Cm)	(D1) Distance in the width direction (Cm)	Sample
0	300	0	0	Undyed
3.925	300	5	1	F1
0	300	0	0	F2
6.28	300	4	2	F3

Where: W_h=3.14×0.25×D1×D2

TABLE 6. Vertical Wicking Test.

Weft Direction			Warp Direction			Sample
$W_{v\ weft}$	(T) Time in Second (Sec)	(D) Distance (Cm)	$W_{v\ warp}$	(T) Time in Second (Sec)	(D) Distance (Cm)	
0.001	300	0.5	0.001	300	0.5	Undyed
0.0023	300	0.7	0.003	300	1	F1
0.0026	300	0.8	0.003	300	1	F2
0.002	300	0.6	0.003	300	1	F3

Where: W_v=D/T

Conclusion

1. A series of novel pyrimidine derivatives and pyridazine derivatives were synthesized by using 2-thioxodihydro-4,6 (1H,5H)-pyrimidinedione as starting new coloured material.
2. The results show that only the wool fabrics was dyed with high shade of yellow colour for all the three coloured materials (F1, F2 and F3).
3. The pH of the solution of the new coloured F2 affected on the colour strength value as well as the increase in dyeing temperature increased the colour strength and best temperature in the range of 85°C-90°C. Also, the dyeing process depended on the concentrations of the dyes (F2).
4. According to radar chart area new coloured materials contribute on improving wool garment performance where it attained a higher radar area comparing with uncoloured sample.
5. A contradictory situation was observed where in spite of acquiring a higher thickness & weight, coloured fabrics achieved a lower bending modulus.
6. The results showed that new materials increased UPF property of wool garment which increase human body protection against harmful sunlight.
7. Depending on wicking test results new coloured material has the possibility to improve moisture transportation in a horizontal & vertical direction of wool samples.

Declaration of conflicting interests

The authors declared that no conflicts of interest with respect to the research, authorship and/or publication of this article.

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