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New natural dyes from red prickly pear produced by addition of metal ions and their application in textile

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Abstract Sicily produces 94% of Italy's farmed crops. The tropical or subtropical plant *Opuntia ficus-indica* (L.) Mill. is cultivated in regions such as the Mediterranean and Central America is used to make the natural dye from the fruits of red prickly cactus pear trees. Where it constructs a good source for food. Betaline pigment extracted and used for synthesis of three new metal complex dyes by addition of bivalent metal ions as copper, cobalt and nickel. These dyes are used for dyeing wool fibers using microwave heating and conventional technique Investigations were conducted into the characteristics of the dyed wool fibers, including dye concentration, pH, color strength, color data, and fastness. The results demonstrated that wool fibres have good colour strength and fastness characteristics. The antibacterial action against a few harmful bacteria and fungus was indicated that the antimicrobial activity of the natural dyes was good results. The metal complex dyes from betaline extracted from red prickly were actively against *Staphylococcus aureus, Pseudomonas aeruginosa, Aspergillus niger* and *Escherichia coli*.

Keywords: Prickly pear, Metal ions, Metal complex, Wool fabric, Antimicrobial activity

Introduction

Opuntia ficus-indica (L.) Mill., the prickly pear cactus, is a tropical or subtropical plant that is grown in regions like the Mediterranean and Central America areas; 94% of cultivated crops of the Italian product is grown in Sicily, where it considered an excellent food source. *O. ficus -indica* is economically important. The cladodes, while scarcely used in modern nutrition and medicine, contain bioactive compounds, well-known for their health-related properties (Pigi, *et al*., 2003). *O. ficus-indica*'s primary ingredient is 80–95% water, with trace amounts of protein (0.5–1%), fiber (1%–2%), and carbs (3–7%). Mucilaginous components with polymers, such as chains of linked β-D-galacturonic acid and R (1-2)-linked L-rhamnose residues, are part of the sugar moiety. (Ali and El-Mohamedy, 2011). Stores sell the edible fruits of most prickly pears under the name "tuna." The pads of prickly pear branches are also prepared and consumed as a vegetable. In retail establishments, they are marketed as "Nopalito." When harvesting or processing prickly pear cacti, extreme caution is needed due to the presence

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of glochids. The prickly pear cactus's fruits and pads are abundant in soluble fibers that are slowly digested and may help stabilize blood sugar levels. The pulp and juice of the fruits are used to make prickly pear nectar (Fernandez-Lopez and Almela, 2001). A natural dye is made from the juice of prickly pears. Fruits were used to isolate betalain pigment. (Ali and El-Mohamedy, 2011). Cactus pears are classified as low-acid foods (pH > 4.5) due to their high pH, which ranges between 5.6 and 6.5 in completely ripe fruits, resulting in a relatively low acidity of roughly 0.05% to 0.18% citric acid. Acidification before heat treatment is therefore necessary to enable pasteurization rather than more stringent sterilization, preserving nutritionally significant ingredients such betalains (Ali *et al.,* 1998). The main organic acid in cactus pear is citric acid (62.0 mg/100 g fruit weight), which is followed by malic acid (23.3 mg/100 g), quinic acid (19.1 mg/100 g), and shikimic acid (2.8 mg/100 g). Oxalic acids are also common. Only trace amounts of isocitric, fumaric, glycolic, and succinic acids were detected (Ali and Abd-Elsalam, 2020). Fruits of many Opuntia species often contain significant levels of ascorbic acid, ranging from 10 to 410 mg/kg. Ascorbic acid levels in the most prevalent 180–300 mg/kg of *Opuntia ficus-indica* (L.) Mill. As a result, cactus pears have higher quantities of ascorbic acid than other common fruits including apples, pears, grapes, and bananas; nevertheless, other vitamins like thiamine, riboflavin, niacine, and carotenoids may only be present in trace amounts. Cactus pear pulp has high quantities of calcium (up to 59.0 mg/100 g) and magnesium (up to 98.4 mg/100 g), while normal levels of sodium, potassium, iron, and phosphorus are found in fruits.

In 2005, Stintzing *et al.* Between 0 and 11.7 betaxanthin:betacyanin cactus pear fruits contain both red betacyanins and yellow betaxanthins, giving them a variety of color tints (Stintzing *et al.,* 2005). It's interesting to note that fruits with betaxanthin concentrations ranging from 10 to 410 mg/k are not yet known to exist in Opuntia species.

The polysaccharide fraction of cactus pear pulp was only recently reported to be composed of a complex mixture of polysaccharides of which less than 50% corresponded to a pectin-like polymer. The hydrocolloid fraction from the fruit pulp of *O. ficus-indica* fruits obtained by ethanol precipitation yielded 3.8% and contained 93.5% sugars. Whereas, after saponification, uronic acid content of 42.3% was determined; neither proteins nor nitrogen were detected. After total hydrolysis, the presence of arabinose, rhamnose, xylose, and galactose in the molar ratio of 1.0:1.7:2.5:4.1 was found. However, further studies are required to completely characterize the hydrocolloid fraction of cactus pear fruit pulp (Rajendhiran *et al.* 2023). Studies on the antimicrobial characteristics of wool fibers that have been dyed with a chitosan-propolis nano composite and painted with a natural dye made from red prickly pears was published (Ali and Abd-Elsalam, 2020). According to (Rajendhiran *et al.* 2023), Prickly pear fruit extract serves as a

sensitizer for dye-sensitized solar cells and a capping ingredient for the sol-gel manufacture of distinct titanium dioxide nanoparticles.

In other cases, prickly pear fruit (opuntia) extract was used to sensitize carotene and anthocyanin-rich flower (hibiscus)/fruit (basella alba, scutia myrtina) extracts, beetroot extract rich in bethanin, and brazilein pigment TiO2 NPs.PPE), which includes betacyanin and indicaxanthin. Because carboxylic acid groups are present, these hues can adhere strongly to the surface of TiO2 NPs. The natural dye from PPE (PP dye) was used to sensitize TiO2 photoanodes, resulting in a 0.5%–1% power-conversion efficiency (Iswariya *et al*., 2017; Prabu and Anbarasan, 2014).

Figure 1. Prickly Pear Cactus *(Opuntia*) fruit

Material and methods

In 2024, this study was conducted at Egypt's National Research Center. El Mahalla Company in Egypt provided wool fibers 10/2. The Samsung M 245 microwave was utilized in this experiment an output of 1,550 watts operating at 245. Reagents of $Cu(NOB_{3B})B_{2B}$, $Co(NOB_{3B})B_{2B}$, $Ni(CHB_{3B}COO)B_2$ were used in the experiment. The red prickly pear plant's fruits were gathered from various sites in the Nobaria region (Beheria governorate, northwest of the Delta, Egypt). In other experiments, the juice of mature fruits was employed as a natural source of colors and antibacterial and antifungal agents against certain microbes. Figure 2 displays the chemical structure of red prickly pear dye made from betaline.

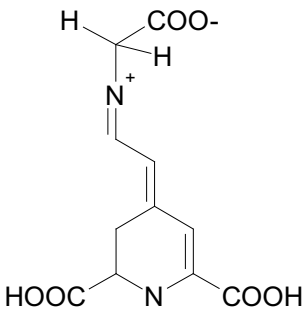

Figure 2. Chemical structure of the coloring component (betaline)

Conventional extraction

Using different concentrations of the dye (2–12%) and time intervals (20–120 minutes), a conventional extraction was performed on 100 milliliters of hot distilled water. The optical density of the dye liquor at 535 nm was determined following filtration and a specific dilution.

Ultrasonic extraction

Using different concentrations of the dye components (2–12%), at different temperatures (50–80 oC), with different sonic powers (100–500W), and for varying durations (20–120 min), ultrasonic extraction was performed in 100 milliliters of distilled water. The dye liquor's optical density was measured at 535 nm following filtering and dilution.

Microwave extraction

Using different concentrations of the dried flower stigmas (0.1 1.5%), microwave extraction was performed in 100 milliliters of distilled water. The dye liquid's optical density was measured at max535 nm following filtering and dilution. For varying lengths of time (1–6 minutes), the dye liquor's optimal concentration at a greater optical density was employed.

Synthesis of metal complex dyes

Syntheses of metal complex dyes weas done by an aqueous solution of the divalent metal ion (Cu^{2+}, Co^{2+}, Ni^{2+}) with the dye extracted from the plant with 1:2 molar ratio was heated for one hour. After cooling the reaction mixture, the complex residue was filtered out. After that, heated ethanol was used to recrystallize the complex. For several hours, the complex was vacuum-dried.

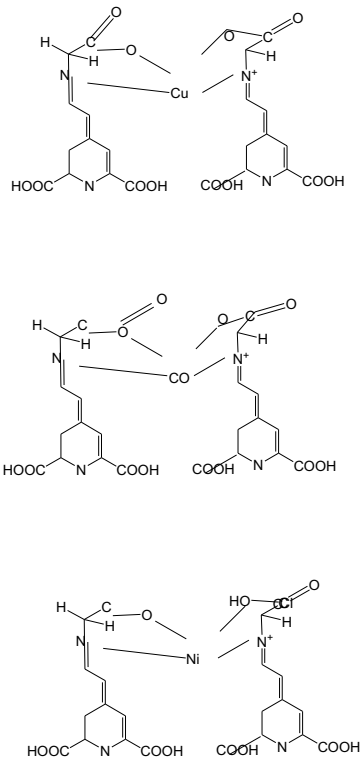

Figure 3. Chemical structure of the metal complex dyes synthesized from betaline

In order to dye wool fibers, a combination of traditional methods and microwave heating was used. Wool fibers with varying pH values (3–7) were dyed for varying lengths of time (1–5 minutes) in a dyeing bath using the colors under examination by microwave and for 60 min by conventional at different conc. (2-8) g/L. After dyeing, wool fibers were rinsed with water and then dried at room temperature. K/S values of dyed wool fibers were measured (Ali *et al*., 2019).

Measurements of color strength (K/S value) was carried out as an Ultra Scan PRO spectrophotometer was used to measure the reflectance of the samples and hence, Spectrophotometric measurements of the K/S were made at wave lengths of λmax 385 nm. The K/S of wool cloth that has been dyed was assessed. where the scattering and absorption coefficients are denoted by K and S, respectively. Using a Hunter Lab Ultra Scan PRO spectrophotometer equipped with a D65 illuminant and a 108 standard observer, the CIELAB coordinates (L* a* b*) of undyed and dyed wool fibers were ascertained (Ali *et al.,* 2019).

Fatness properties measurements

Fastness property measurements were conducted using ISO standard procedures. Specific tests for They were ISO 105X12 (1987), ISO 106-C06 (1989), ISO 105-E04 (1989), and ISO 105-B02 (1989) for color fastness to rubbing, washing, perspiration, and light. The samples' color variations were evaluated using a precise Gray scale.

Assessment of the antimicrobial activity

Peptone 5.0 g, beef extract 1.5 g, yeast extract 1.5 g, NaCl 5.0 g, and agar 20 g at pH 7.5 made up the nutrient agar culture (g/L), which was prepared and autoclaved at 121 °C for 20 minutes. Nutrient agar of uniform thickness was made in sterilized petriplates. Gram-positive bacteria like Bacillus subtilis and Pseudomonas aeruginosa, as well as gram-negative bacteria like Escherichia coli, were among the species that were cultivated overnight at 37 °C and 120 rpm in 2 mL of nutritional broth.

The agar plates were seeded using this soup. Under sterile conditions, wool samples were placed on top of the seeded medium. The zones of inhibition were assessed following an overnight incubation period at 37 °C. Samples of undyed wool were measured in the second series of studies (Dhanalakshmi *et al.,* 2013).

Results

Elemental analysis and the color of complexes

Elemental analysis was measured to evaluate the % of C, H and N for the prepared metal complex dyes from betaline extracted from red prickly pear plant as shown in Table 1 which confirmed the structure of the synthesized dyes.

Table 1. Elemental analysis and the reaction period and color of complexes

Complex	**No.**	**Color**	**Analysis of Elments**					
			% C		% H		**% N**	
			Calculated	Found	Calculated.	Found	Calculated.	**Found**
$[Co(B)_2]$.$2H_2O$	1	Pink	54.5	54.1	3.3	3.0	11.6	**11.2**
$Na_2[Ni(AR151)_2]$.$2H_2O$	2	Brown	54.5	54.1	3.3	3.1	11.6	**11.3**
$Na_2[Cu(AR151)_2]$	**3**	**dark red**	**56.4**	**56.0**	**3.0**	**2.8**	**11.9**	**11.3**

The infrared data were indicated the chemical structure of the dyes under investigation as shown in Table 2. Complexation reaction with CO^{2+}, CU and Ni ions were conducted by a metal-ligand-betaline interaction, where the betaline structure is depicted in Figure 2, and only the -OH functional group was involved in the coordination reaction. They had described the synthetic dyes. through infrared spectroscopy.Performance evaluation of these dyes were applied on wool fibers. Dye structure was confirmed by IR.

Table 2. Infrared data for the dye and its copper, cobalt, and nickel complexes obtained are as following

Compound	**Infrared data (cmP^{-1P})P^{Ap}**					
	υB_{OHB}	υB_{NHB}	$\upsilon B_{C=NB}$	$\upsilon B_{C=CB}$	υB_{M-OB}	**υB_{M-NB}**
Coloring component	3390 (m)	-	-	1597(m)	-	**-**
Dye 1	3385(m,b)	-	-	1596(m)	573(m)	**498(w)**
Dye 2	3401(m,b)	-	-	1594(s)	572(m)	**474(w)**
Dye 3	**3400(m,b)**	**-**	**-**	**1593(m)**	**525(m)**	**499(m)**

P^{aP} s, strong; m, medium; b, broad

Effect of concentration on dye amount

The coloring component's relative extractability at varying concentrations (2–8 g/l) was assessed using conventional heating for an hour at the boiling temperature and microwave heating for five minutes. The absorbance of the dye is higher for microwave than conventional as shown in Figure 4. It was also observed that the highest absorbance obtained at 3g/L for conventional dyeing and at conc. 5g/L for microwave.

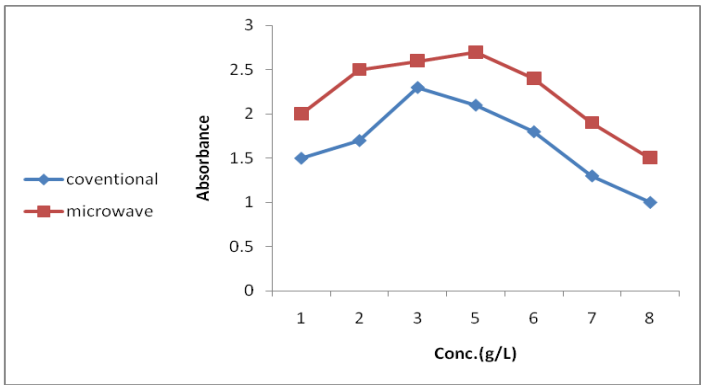

Figure 4. Impact of concentration on the dye amount

Impact of extraction duration

Betaline dye was extracted using microwave heating at a concentration of 4g/l over a range of time intervals (1–7) minutes. According to the results obtained, dye extraction by microwave heating was higher than conventional method as well as the highest values attained after five minutes (Figure 5). This is explained by the intense movement in the liquor caused by the wave guides in microwave irradiation. Any material can pass through the uniform exposure provided by the resulting microwave distribution.

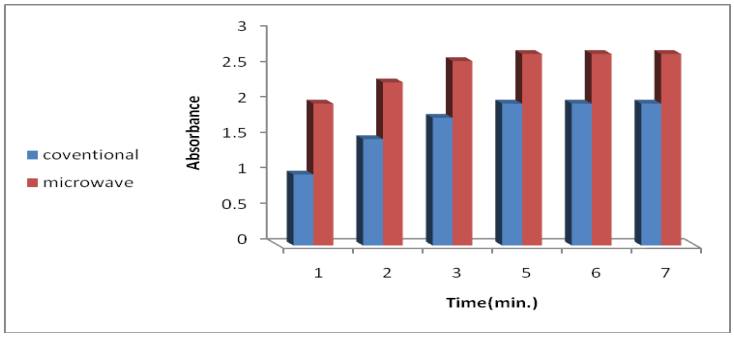

Figure 5. Effect of extraction Time using microwave and conventional techniques on the absorbance

Effect of dye bath pH

The results demonstrated that the dyeing ability of wool fibers with the produced complex dyes is significantly influenced by the pH values of the dye bath. The relationship between wool fabric and dye structure explains the impact of the dye bath pH. When the dye's complex anion was linked to the fiber by ionic forces, the ionic attraction enhanced the fiber's dye capacity, increasing the color strength (K/S) and the maximum value of K/S. K/S was achieved at pH 5 as clearly observed in Figure 6.

The impact of temperature

The effect of temperature on the dyeing capabilities of wool fibers with complex dyes at various temperatures (30-90 ^{o}C) is illustrated in Figure 7. It is evident that as the dyeing temperature rose, the color strength increased for peaking at 90 degrees Celsius.

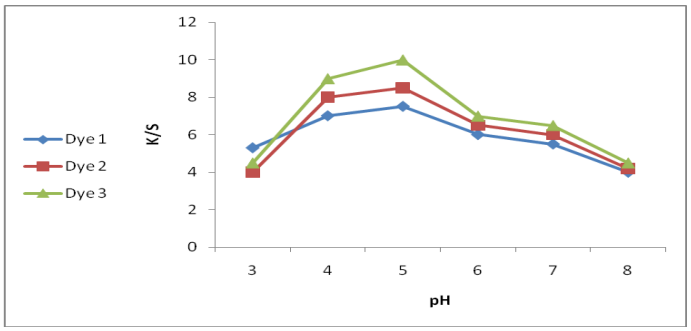

Figure 6. The impact of dye bath pH on wool fabric color strength. Conditions for dyeing: 2% shade, 40:1 lacquer ratio (LR), 90 °C, 40 minutes.

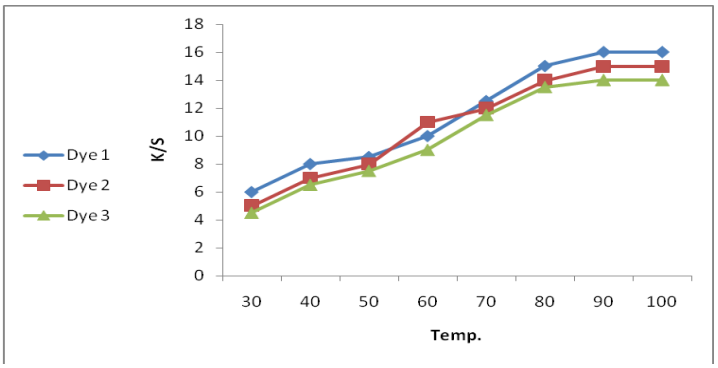

Figure 7. impact of the dyeing temperature on the wool garments' color intensity. 2% shade, L.R. 40:1, pH 4.5, and 40 minutes were the dyeing conditions. 1% salt conc

Impact of dye concentration on dyeing procedures

The synthesized dyes were applied to wool garments at varying concentrations (2–10 g/l) of a liquor ratio of 1:100. Conventional heating methods, such as boiling for an hour and heating in a microwave for five minutes, were used to dye wool cloth. The findings demonstrated that using the usual approach, increasing the dye concentration to 10g/l raised the K/S. When using the microwave technique, the K/S rose to 10 g/l of dye. It has been discovered that microwave dyeing works better than traditional dyeing as shown in Figure 8.

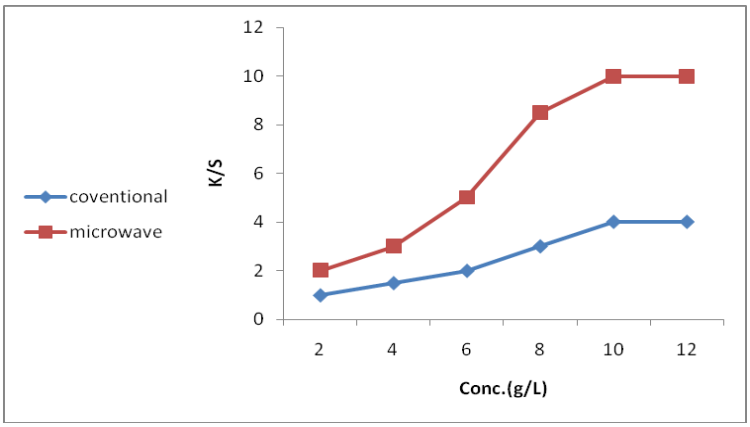

Figure 8. Impact of dye conc, on dyeing process

Dyeing time's impact on dyeing procedures

Additionally, the impact of dyeing time was examined. How dying time affected color concentration is shown in Figure 9. When employing either microwave method, it was found that the color strength rose with an increase in dyeing time. When using the conventional approach, it took 50 minutes to produce the same effect, however the microwaves' effect grew as the dyeing time increased up to 5 minutes (Figure 10).

The levelness of the colored fiber was greatly enhanced by microwave heating. When conventional heating is used, the absorbed dye is concentrated on a small number of peripheral regions and dispersed unevenly on individual fibers. A higher level of dye penetration resulted with the application of microwave heating. The dyestuff was more evenly and highly concentrated on the fiber surface after microwave heating, which allowed it to diffuse into the fiber's interior.

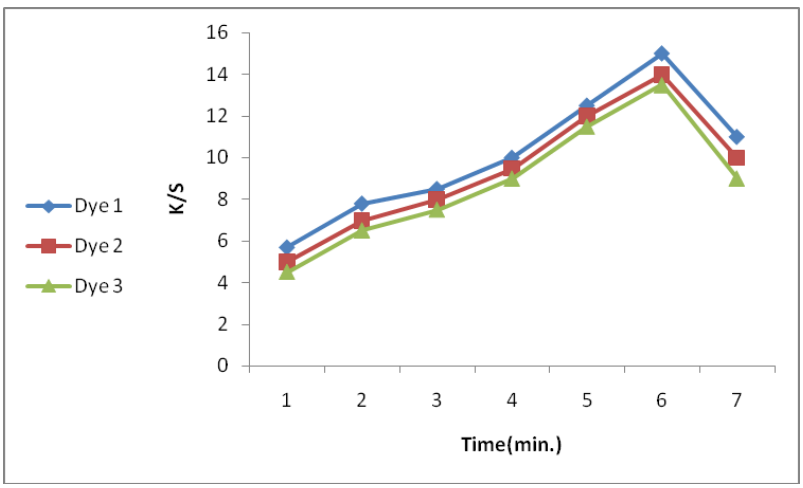

Figure 9. Effect of dyeing time on dyeing processes using microwave

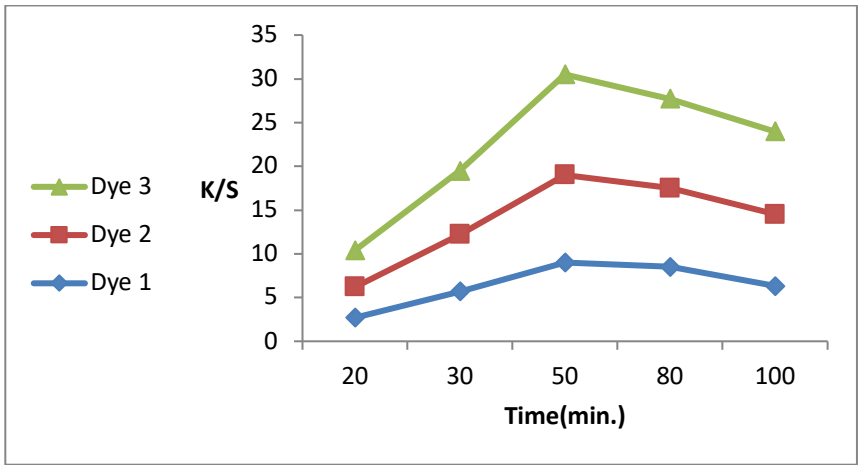

Figure 10. Effect of dyeing time on dyeing processes using conventional

Fastness properties for wool fabric dyed by microwave technique

The wool fabric samples' capacity to retain color after dying and how it interacts with fibers were shown in Table 3. Result displayed the dyed fabrics' fastness characteristics. The outcomes showed that the colored samples have good fastness qualities. Using a grey scale, the fastness of microwave radiation as a heating source was assessed with respect to rubbing, washing, sweating, and light fastness. The outcome demonstrated that the wool fiber, when exposed to microwave radiation, was more resilient for washing and sweating and exhibited a higher color intensity. It might be linked to a rise in dye penetration.

Table 3. Test of fastness characteristic for wool samples using the synthesized complex dyes by microwave technique

Dye	Fastness to rubbing		Fastness to washing fastness			Fastness to perspiration						Light fastness
						Alkaline			Acidic			
	Dry	Wet	Alt	Sc	Sw	Alt	Sc	Sw	Alt	Sc	Sw	
1	4	5	4	5	4-5	5	3-4	4	4-5	4	5	7
2	4	3-4	5	4-5	4	4-5	4	4-5	4	4	4-5	6
3	5	4	4	4	4-5	4	4-5	4	4	4	5	6

Note: Alt: Alteration, Sc: Staining on cotton and Sw: Staining on wool

Fastness properties for wool fabric dyed by conventional technique

The dyed fabrics' fastness characteristics is shown in Table 4. The results showed that the dyed samples had good fastness qualities.

Table 4. Fastness properties of the dyed wool fabric by convention anal technique

Dye	Fastness To rubbing		Washing fastness			Fastness to perspiration						Light fastness
						Alkaline			Acidic			
	Dry	Wet	Alt	Sc	Sw	Alt	Sc	Sw	Alt	Sc	Sw	
1	4	4	4	5	4	5	3	4	4-5	4	5	6
2	5	3	5	4	4	4-5	4	4-5	4	4	4	5
3	4	4	4	4	4-5	4	4	4	3	4	4	5

Alt: alteration Sc: staining on cotton, Sw: staining on wool

Antimicrobial activity

It clearly shown that the antibacterial activity of wool fibers is influenced by dye concentration. Different bacterial and fungal strains showed varied levels of antimicrobial activity. The result showed that the colored fibers showed more antimicrobial than uncolored fibers. Result showed the metal complex dyes from betaline extracted from red prickly pear were antimicrobial activities against *Staphylococcus aureus, Pseudomonas aeruginosa, Aspergillus niger* and *Escherichia coli* (Table 5).

Table 5. Antimicrobial activity of dyed wool by metal complex dyes from betaline extracted from red prickly pear

Microbe	Growth reduction (%) of several microorganisms on fibers at varying dye concentrations under investigation					
		0 control	**1**	**1.5**	**2**	**2.5**
Staphylococcus aureus **G+**	**Dye 1**	14	20	47	54	**89**
	Dye 2	12	28	29	53	**90**
	Dye 3	14	22	32	55	**87**
Pseudomonas aeruginosa	**Dye 1**	20	44	50	56	**80**
	Dye 2					
	Dye 3	16	30	35	46	**74**
Aspergillus niger	**Dye 1**	20	41	52	60	**78**
	Dye 2					
	Dye 3	17	39	48	55	**65**
Escherichia coli **G-**	**Dye 1**	11	26	59	60	**75**
	Dye 2					
	Dye 3	8	16	27	40	**60**

Discussion

Microorganisms that cause infections, offensive odors, color deterioration, and textile damage are common in textile items. Antimicrobial fabrics can be used to make a wide range of products, such as sportswear, outdoor apparel, undergarments, shoes, furniture, hospital linens, towels, wipes, and wound care wraps. To assess the effectiveness of antimicrobial textiles, a number of test techniques have been devised, including the dynamic shake test (a quantitative method similar to serial dilution and the plate count method) and the agar diffusion test (a qualitative method similar to the halo method) (Ben-David and Davidson, 2014). The antibacterial effectiveness of wool fibers is colored with red prickly pear dyes was evaluated using One harmful fungal isolate and two pathogenic bacterial isolates, Staphylococcus aureus and Escherichia coli, *Aspergillus niger*. It was investigated antimicrobial active by using various dye concentrations when to the applied dye wool. It demonstrated that all tested microorganisms were larger inhibitory zones in dyed wool due to increase the dye concentration than the undyed wool. With a 2% concentration showed high inhibitory zone. It suggested that the color derived from prickly pear dyes was a very potent antibacterial against the tested microorganism, *E. coli* and *S. aureus.*

According to Fernandes Júnior *et al.* (2005), metal complex dyes have a variety of antimicrobial mechanisms, such as preventing cell division, breaking down slowing protein synthesis, blocking bacterial motility, deactivating enzymes, bacteriolysis, and microbial cytoplasmic cell membranes and cell walls. Through the formation of hydrogen and ionic connections, petaline interacts with a variety of microbial proteins, changing their three-dimensional (3D) structure and, ultimately, their functioning. (Wink, 2008, Wink, 2015). The antimicrobial activity towards some species of bacteria and a fungus was also determined and the results obtained showed high percentage reduction for dyed wool fabric with the three metal complex dyes.

Three metal complex dyes were synthesized using a new natural dye that was derived from prickly pear juice. These dyes were then used as natural wool dyes with high dye absorption and good fastness characteristics. Wool fibers dyed with these dyes were tested for antimicrobial activity using diffusion agents. The samples showed a strong inhibitory zone, according to the findings of tests using E. Coli, B. subtilus, Pseudomons aeruginosa, and Staphylococcus aureus (Ali and Abd-Elsalam, 2020).

Our research demonstrates the ability of the natural red prickly pear dyes to produce vivid colors and color fastness. In the future, they might be a significant supply of raw materials. Since it could significantly help in the creation of color molecules, the chemical alteration of natural substances like the dye in issue could be an intriguing area of research. Samples of wool cloth dyed with microwave irradiation as a heating source were tested for color fastness. using a grey scale. The fastness against rubbing, washing, perspiration, and light fastness were all considered (Ali and El- Khatib, 2010).

The outcome demonstrated that the wool fiber, when exposed to microwave radiation, is more resilient to washing and sweating and exhibits a higher color intensity. It may be attributed to increase dye penetration and its interaction with fibres (Ali, and Abd- Elsalam, 2020). The data obtained indicate that the red prickly colorant one possible source of natural food coloring (Kanner *et al.,* 2001) In our study, wool is dyed with metal complex dyes synthesized from betalain pigment, which is produced from red prickly pears. These dyes are stable in the pH range of 4-5.

Conflicts of interest

The authors declare that there are no conflicts of interest regarding the publication of this paper.

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