Investigation of the Printability of Henna Before and After Miniaturization on Natural Fabrics

A.A. Ragheb¹, S. Tawfik², J.I. Abd El-Thalouth², and M.M. Mosaad²

¹Textile Research Division, National Research Centre, Cairo, Egypt

²Faculty of Applied Arts, Helwan University, Cairo, Egypt

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ABSTRACT: A novel nanoscale henna natural dye with particle size less than 100 nm were successfully prepared by using ultrasonic stirrer. Henna natural dye as ecofriendly dye was studied to clarify the impact of nature of nano-size color particles on size, shape, and particle distribution of the natural dye. The work was extended to study the K/S and overall color fastness properties of the printed natural fabrics (wool, silk and cotton) in presence and absence of mordant. Results showed that the K/S values of nano scale samples acquire higher values when compared to that of the original samples, irrespective of the nature of the fabric used. Mordant for example Alum that incorporated with original henna can be omitted, and substituted by nano-henna without mordant on printing silk and cotton fabrics. Results also shows that the pre-mordanting acquired K/S values higher than the simultaneous mordanting irrespective of the kind of fabric used, or dye particles size used, or henna concentrations. While on using tannic acid as a mordant, color fastness to rubbing, and perspiration properties of nano dye is found to be better than that of the original.

KEYWORDS: Textile, Printing, Nanotechnology, Henna dye, natural fabrics.

1 INTRODUCTION

Worldwide, growing consciousness about organic value of eco-friendly products has generated renewed interest of consumers towards use of textiles (preferably natural fiber product) dyed or printed with eco-friendly natural dyes. Natural dyes are known for their use in coloring of food substrate, leather as well as natural fibers like wool, silk and cotton as major areas of application since pre-historic times⁽¹⁾.

On the hand, based on their origin, natural fibers can also be classified as cellulosic (from plants) like cotton⁽²⁾; and protein (from animals) like wool^(3,4)or silk⁽⁵⁾.

The word 'natural dye' covers all the dyes and pigment derived from the natural sources like plants, animal and minerals. Natural dyes can be used to dye different natural and man-made materials ⁽⁶⁾.

Textile coloration using natural dyes found to yield poor color, have inadequate fastness properties. To overcome such hassle, mordants are used. Metal ions of mordants act as electron acceptors for electron donors to form co-ordination bonds with the dye molecule⁽⁷⁾. Because of the chemistry associated with dyes from natural materials, it is necessary to utilize fibers, which have dye sites that can bond molecularly with these dyes⁽⁸⁾. Cotton has no inherent affinity for most natural dyes. However the affinity of cotton can be modified to make it dyeable with natural dyes by the use of metallic salts mordants) or the process of cationization which creates positively charged sites on cotton or by addition of NaOH or enzymes⁽⁹⁾. Further, these dyes can directly dye protein fabrics under acidic pH since they have basic amino groups in the same manner as synthetic acid dyes.

The word Henna has its origin in the Arabic word Al-Hinna. In botanical terms it is, Lawsoniainermis, commonly known as Mehdi/Mehandi is a shrub or small tree frequently cultivated in India, Pakistan, Egypt, Yemen, Iran and Afghanistan. Henna is

an ancient dye, evidence being the Egyptian mummies found in the tombs that had their nails dyed with henna. It is also used in many countries for dyeing hair, eyebrows and fingernails during religious festivals and marriages etc. the powdered leaves of this plant (aqueous paste) are used as a cosmetic for staining hands, palms, hairs and other body parts. Nowadays it is used to gardening as well as fabrics to dye including silk, wool, and leather. The dye component present in the leaves of henna is "Lawseone" Lawsoniainermis is belonging to the familyLagthraceais identified as 2 hydeoxel 1,4, naphthoquinone. with Color Index Number 75480; Natural Orange 6^(10, 11).



Scheme (1): Coloring component of henna (L. inermis) leaves -Lawesone.

Henna is an organic red pigment, yellow crystals. It originates from the primary glucosides, the hennosides, in the course of drying of the leaves and the oxidation of a glycone thus liberated. The leaves also contain flavonic pigment (luteoline) of yellow color and tannins (6% of gallic acid) which play the role of organic mordant ⁽¹²⁾. Henna is consider as a disperse dye, and has Antibacterial properties of henna dyed wool fabrics against E. coli and S. aureus are also reported ^(11, 13).

The urgent need for innovative technology, as alternative for the conventional production process, for achieving lower cost, cleaner production process and products, as well as attaining improved lifetime, better quality and performance properties is actually a global necessity. Nanotechnology occupies a top position among frontier technologies that responds to this need.Nanotechnology is the science and technology of designing, constructing and creating of novel nano-scale structure, 1nm to 100 nm in size, with huger quality, novel performance properties, along with fewer defects compared with those of the bulk material.

The present work is undertaken with a view to harness nanotechnology as one of the most important frontier for development printing natural fabrics using eco-friendly natural color namely henna. Role of miniaturization of the used natural dye on their printability of wool, silk and cotton in presence and absence of mordant will be investigated. Besides comparative studies of the K/S and overall color fastness properties of the printed goods before and after miniaturization will be done.

2 EXPERIMENTAL

2.1 MATERIALS

Fabrics

- Cotton fabric:Mill desized, bleached and mercerized cotton fabrics 165 g / m² produced by Misr/Helwan for Spinning and Weaving Company, Helwan, Egypt.
- Wool fabrics: Mill scoured 100% wool fabric supplied by Misr Company. for spinning and weaving (Mehalla El-Kubra) 210 g / m², Mehalla, Egypt.
- Silk fabrics: Mill scoured natural Silk fabric of plain weave 60 g / m² supplied by El-Khateib Company, Souhag, Upper Egypt.

Dyestuff: natural Coloring substance turmeric (Henna) [which have been purchased from local market] was extracted according to the procedure described latter.

Thickening agents: Commercial synthetic thickening agent, namely Printofixthickener MTB 01 EG liq manufactured by CLARIANT company was also used.

Mordants: Alum [hydrated double sulfate of potassium hydrogen sulfate (KAI (SO₄)₂.12 H₂O], and tannic Acid.

2.2 METHODS

2.2.1 EXTRACTING OF NATURAL COLORING MATTER

100 g. henna dry powder were added to 1000 ml. water and subjected to boiling under reflux for 30 min. The mixture was left to cool at room temperature and then filtrated off. The filtrated solution was concentrated using a laboratory Rotavapour.

2.2.2 PREPARATION OF NANO PARTICLES OF HENNA NATURALDYE USING ULTRASONIC STIRRER

Different amount "X" (3, 5, or 7gm) of filtrated solution of Henna were suspended in 100ml distilled water under stirring then the solution were set to motion in the Ultrasonic stirrer (The probe is turned to resonate at specific frequency, 20 KHZ \pm 100 HZ). The Ultrasonic stirrer was operated for 60 min at ca 80°C to reach the nano-size.

2.2.3 PREPARATION OF THE PRINTING PASTES

The extracted henna color was subjected to minimization using Ultrasonic stirrer as previously mentioned. Different printing pastes containing natural henna color before and after minimization were prepared according to the following recipes:

| Dye suspension original or nano sample * | 20 g |
|--|------|
| Urea | 2.5g |
| Thickener | 2.5g |
| Binder | 5g |
| Sodium dihydrogen phosphate dehydrate | 0.5g |
| Mordant ** | Х |
| Distilled water | Y |
| Total | 100 |

* 20g was taken from each dyeing solution (original or nano-size) containing henna color at a concentration of 3, 5, or 7 g dye in 100 ml water.

** The mordant was incorporated direct in the printing paste.

2.2.4 MORDANTING OF NATURAL FABRICS

Mordanting of the natural fabrics (cotton, wool or silk) were conducted using two different techniques; either premordanting or simultaneous i.e. added directly to the printing paste were used.

2.2.5 PRINTING TECHNIQUES

The aforementioned three different fabrics, i.e. wool, cotton, or silk were printed with the prepared printing pastes via screen, i.e. screen printing technique.

After printing and drying the printed goods were subjected to steaming at 115°C for 10 minutes for silk, and for 20 minutes for both cotton and wool followed by thoroughly washing and finally air-dried. At the end, the fabrics were assessed for K/S and overall fastness properties.

2.3 TESTING, ANALYSIS AND MEASUREMENTS

2.3.1 TRANSMISSIONELECTRONIC MICRO-SCOPY (TEM) ⁽¹⁴⁾

Particle shape and Size were obtained using aJEOLJEM 1200. Specimens for TEM measurements were prepared by dissolving a drop of colloid solution on a 400 mesh copper grid coated by an amorphous carbon film and evaporating the solvent in air at room temperature. The average diameter of the natural dye nanoparticles was determined from the diameter of 100 nanoparticles found in several arbitrarily chosen areas in enlarged microphotographs.

2.3.2 COLOR STRENGTH AND FASTNESS

The color strength (K/S) of the samples was evaluated by light reflectance technique using Shimadzu UV/Visible spectrophotometer⁽¹⁵⁾.and the color overall fastness properties: i.e. to washing, perspiration or rubbing fastness were assessed according to standard methods⁽¹⁶⁾.

3 RESULTS AND DISCUSSION

3.1 DEPENDENCE OF THE SIZE OF HENNA PARTICLES ON ITS CONCENTRATION

To start with TEM investigation of the particles size of the henna color, Figures 2, 3 and 4 show the TEM micrographs of henna at original size, i.e. before subjecting to ultrasonic stirring at a concentration of 3, 5 and 7%. Figures 5, 6 and 7 represents the particle size after subjecting to ultrasonic stirring for 60 min, at Ca 80 °C.



Fig (2): 3% original henna

Fig (3): 5% original henna





Fig (5): 3% nano henna

Fig (6): 5% nano henna

Fig (7): 7% nano henna

A close examination of figure 2 which represent the TEM at a concentration of 3% before subjecting to ultrasonic stirring signifies an average particle size of 103.7 nm. Indeed this average covers a wide distribution of size particles ranging from 56.9 to 131.9 nm. While figures 5 represents TEM micrograph of the same sample after subjecting to ultrasonic technique. Figures 3,4 and figures 6, 7 represent the TEM before and after miniaturization at concentrations of 5 and 7 % respectively.

While table (I) summarize the effect of concentration of henna on the particle size before and after nonfiction.

Table I: Comparison between henna particle size before and after miniaturization using ultrasound based technique

| | | Original size | | | | | | |
|-------|----------------|------------------------------------|------------------------------------|---------------------------------|------------------------------------|---------------------------------|------------------------------|---------------------|
| Dye | Con. of dye | Average size of dye particle | Max. size of dye particle | Min. size of dye particle | Average size of dye particle | Max. size of dye particle | Min. size of dye particle | Percent decrease |
| | 3% | 103.7 | 131.9 | 56.9 | 28.1 | 56.5 | 9.16 | 72.90% |
| Henna | 5% | 223.9 | 251.1 | 215 | 69.2 | 131.5 | 46.6 | 70.41% |
| | 7% | 377.5 | 470 | 290 | 102.9 | 153.3 | 45.3 | 72.74% |

Conditions: Time of stirring 60 min, temperature of stirring ca 80°C.

It is clear from the micrographs that the size, shape and particle size distribution of nano-dye rely on dye concentration.

It is also clear that by Increasing the dye concentration from 3 to 7gm/100ml water increases the average of dye particle size from 28.1 nm to 102.9nm at nano-form, and from 103.7nm to 377.5nm at original form. With greater tendency of the nanoparticles to aggregate / agglomerate.

The decrease percentage of miniaturization of particle size are (72.90%, 70.41%, 72.74%) at dye concentrations' (3, 5, and 7) respectively.

There is also noticeable gradual increments in the nanosized henna particles by increasing the henna concentration within the range studied. This suggest that increased amounts of henna in cases of its higher concentrations seem to protect its particle sizes from the action of mechanical and thermal energies involved in miniaturization. Once this is the case, the protection would be remarkable at higher henna concentration.

3.2 EFFECT OF MINIATURIZATION OF HENNA COLOR TO NANO-SIZED ON ITS PRINTABILITY ON NATURAL FABRICS

To investigate the effect of decreasing the particle size of henna to the nano-form on its printability on natural fabrics, printing pastes containing the aforementioned three concentrations before and after miniaturization were prepared in presence or absence of mordants. The prepared pastes were applied to print the aforementioned three natural fabrics according to the procedure described in the experimental section. Given below the results obtained along with the appropriate discussion.

3.2.1 COMPARISON BETWEEN ORIGINAL AND NANO-SIZE: (IN ABSENCE OF MORDANT)

Table II represents the data of K/S obtained using different concentrations of henna before and after miniaturization.

| Fabric | Due Con % | Without mordant | | | | | | |
|--------|------------|------------------|--------------|------------|--|--|--|--|
| Fabric | Dye con. % | In original Form | In nano Form | % increase | | | | |
| | 3 | 1.85 | 1.98 | 7.03% | | | | |
| Wool | 5 | 2.44 | 2.84 | 16.39% | | | | |
| | 7 | 2.50 | 2.94 | 17.60% | | | | |
| | 3 | 1.62 | 1.65 | 1.85% | | | | |
| Silk | 5 | 2.04 | 2.58 | 26.47% | | | | |
| | 7 | 2.29 | 3.12 | 36.24% | | | | |
| | 3 | 0.82 | 1.08 | 31.71% | | | | |
| Cotton | 5 | 1.50 | 1.81 | 20.67% | | | | |
| | 7 | 2.04 | 3.18 | 55.88% | | | | |

Table II: Effect of miniaturization of henna color to nano-sized on K/S in absence of mordant

It is clear from the data of Table II that the K/S is oppositely related to the size of the nano particles. Decreasing the particle size is accompanied by substantial enhancement in color strength.

The increase in K/S on decreasing the particle size to the nano-scale may be due to the virtue of their small nano-scale size and large surface area which make the particle defuse faster in the interior of the printed fabrics and distributed themselves on the fabric surface during printing. In so doing, they produce prints with strong color shade and, therefore, higher color strength compared with the original particles which acquire relatively larger size.

In addition, as the concentration of the henna dye increases, the K/S value increases irrespective of the particle size either (original or nano size). For example in original-form the increase of K/S from 1.85 to 2.44 to 2.50, while in nano-form increases from 1.98 to 2.84 to 2.94.

It is clear from the data that irrespective of the fabric used; the K/S values of nano samples are higher than original samples. Moreover, the highest K/S was obtained at 7% of henna concentration in both original and nano-form.

Proteinic fabrics i.e. wool and silk acquire high affinity for natural colors than cellulosic fiber.

3.2.2 COMPARISON BETWEEN ORIGINAL AND NANO-SIZE OF HENNA DYE IN PRESENCE OF PRE-MORDANT WITH (ALUM OR TANNIC ACID)

Irrespective of the kind of fabric used; the K/S values of nano printed samples are higher than original samples, this phenomenon holds true in order to a matter is reduced in size, it changes its characteristics, such as color and interaction with other matter. Moreover, the highest K/S values obtained on using a concentration of 7% of henna dye.

Table III represent the effect of nature of mordant (Allum or tannic acid) on the K/S of the fabrics printed with henna dye before and after miniaturization.

Table III: Effect of miniaturization of henna color to nano-sized on K/S in presence of (Allum and Tannic acid) as a mordant

| | Due Con | | With Allum | | With Tannic acid | | | |
|--------|---------------|---------------------|-----------------|------------|---------------------|-----------------|------------|--|
| Fabric | bye Con. % | In original Form | In nano Form | % increase | In original Form | In nano Form | % increase | |
| | 3 | 2.28 | 2.62 | 14.91% | 2.71 | 3.08 | 13.65% | |
| Wool | 5 | 2.42 | 2.93 | 21.07% | 3.85 | 3.96 | 2.86% | |
| | 7 | 2.44 | 3.96 | 62.30% | 4.23 | 5.99 | 41.61% | |
| | 3 | 1.46 | 1.51 | 3.42% | 4.00 | 4.39 | 9.75% | |
| Silk | 5 | 1.88 | 2.32 | 23.40% | 4.42 | 4.76 | 7.69% | |
| | 7 | 1.92 | 3.84 | 100% | 4.71 | 4.98 | 5.73% | |
| | 3 | 0.85 | 0.96 | 12.94% | 1.88 | 2.05 | 9.04% | |
| Cotton | 5 | 1.69 | 1.79 | 5.92% | 2.65 | 2.70 | 1.89% | |
| | 7 | 1.94 | 2.58 | 32.99% | 2.82 | 4.38 | 55.32% | |

The former results are referred to the influence of sonication of dye that leads to particle size reduction. Grinding increases the specific surface area (ssa) of the ground particles due to particle size reduction ⁽¹⁷⁾. A feasible technique for particle-size reduction is ultrasound. Cavitational collapse sonication in solids leads to micro-jet and shockwave-impacts on the surface, together with inter-particle collisions, which can result in particle-size reduction ⁽¹⁸⁾.

Alum, (Aluminum potassium sulfate), is the most common aluminum-based mordant used in dyeing cellulose fibers. Metal complex formation between dyes and Al^{3+} is shown in Scheme(2) The connection between fiber and metal- dye complex is illustrated in Scheme(3). Alum usually refers to a hydrated double sulfate of potassium hydrogen sulfate (KAl(SO₄)₂.12 H₂O), which confers evenness and brightness on dyed fabrics⁽¹⁹⁾.



Scheme (2): Nature of bonding between a natural dye and AI^{3+}



Scheme (3): The connection among cellulose, mordant and natural dye

Another agent used to improve the color fastness of natural dyes on textiles is tannic acid.

Tannic acid or tannins are used as a primary mordant for cotton and cellulosic fibers as they do not have much affinity for metallic mordants. A cotton fabric treated with tannic acid can absorb all types of metallic mordants. Tannic acid forms a color lake with dye inside the fiber which fixes dye to fibers more strongly.



Scheme (4): Structure of Tannic acid

Tannins form following three type of bonds with proteins (eg., wool and silk) and cellulose fibers.

- 1. Hydrogen bonds between the phenolic hydroxyl groups of tannins and both the free amino and amido groups of proteins.
- 2. Ionic bonds between the suitably charged anionic groups on tannin and cationic groups on protein.
- 3. Covalent bonds formed by the interaction of any quinine or semi-quinone groups in the tannins and any other suitable reactive groups in the protein or other polymer.



Scheme (5): Reaction of protein (wool/silk) petide chain with natural tannins



Scheme (6): Reaction of cellulose with natural tannins

3.2.3 COMPARISON BETWEEN NANO-DYE AND ORIGINAL DYE WITH MORDANT

To investigate the possibility of using nano-dye instead of using mordants with original dye. Different samples (wool, silk or cotton) were printed with different mordants (alum or tannic acid) incorporated with original henna dye, while other samples printed with only nano-henna dye for comparison.

| Nature of | Dye conc. | K/S of Nano | K/S of original dye incorporate with mordant | | | |
|-----------|-----------|-------------|---|-------------|--|--|
| Tabric | 70 | aye | Alum | Tannic acid | | |
| | 3% | 1.98 | 2.00 | 2.46 | | |
| Wool | 5% | 2.48 | 2.06 | 2.95 | | |
| | 7% | 2.94 | 3.17 | 3.09 | | |
| | 3% | 1.65 | 1.20 | 2.27 | | |
| Silk | 5% | 2.58 | 1.45 | 2.71 | | |
| | 7% | 3.12 | 1.94 | 3.17 | | |
| | 3% | 1.08 | 0.94 | 1.66 | | |
| Cotton | 5% | 1.81 | 1.41 | 2.14 | | |
| | 7% | 3.18 | 2.37 | 2.96 | | |

Table (IV): comparison between printing dye in nano-form, and dye in original form incorporate with mordant

It is clear from the data of table IV that; Nano-henna dye could be used successfully instead of the original dye incorporated with Alum mordant when applied on silk and cotton fabrics. The K/S values of the nano-henna acquired higher than original dye incorporated with Alum mordant when applied on silk and cotton fabrics. For example K/S increases from 1.20 to 1.65, from 1.45 to 2.58 and from 1.94 to 3.12 with using henna at concentrations (3, 5, and 7%) on silk in (original henna incorporated with alum) and nano-henna respectively.

3.2.4 Comparison between original-dye, nano-dye, and nano-dye incorporated with nano-tannic acid at dye concentration 5%

3.2.4.1 ON USING TANNIC ACID MORDANT

It is clear from figure (8)that, irrespective of the fabric used, the dye and Tannic acid mordant in nano-form acquired the highest K/S values, followed by the dye in nano-form, while the dye in original form acquired the lowest values.



Fig. (8):Comparison between K/S of henna dye in original and nano- size and nano-dye incorporate with nano-tannic acid

For example, the K/S of the silk samples was 2.04, 2.58 and 3.45 for original-form, nano-form, and (dye and mordant) in nano-form respectively. In case of the wool the data represent that, the dye in nano form has slightly increase in K/S.

While in case of Cotton samples, Cotton has no inherent affinity for most natural dyes, as previously reported ⁽⁹⁾.

It is clear that the nano-form of the dye incorporate with nano-tannic acid gives the highest percent increase of K/S by 30% than original form and by 7.73 % than nano-dye.

3.2.4.2 ON USING ALUM MORDANT

The same trend as tannic acid was obtained with using Alum on silk and cotton fabrics. While using on wool fabrics the nano-henna acquired the highest K/S values irrespective the dye concentration used, as shown in table V

| | | K/S of | | | | | | |
|---------------------|-----------|---------------------------|-----------------------|-------------------------------------|--|--|--|--|
| Nature of fabric | Dye conc. | Henna in original form | Henna in nano form | (Henna and Alum) in nano form | | | | |
| | 3% | 1.85 | 1.98 | 2.16 | | | | |
| Wool | 5% | 2.44 | 2.48 | 2.30 | | | | |
| | 7% | 2.50 | 2.94 | 2.81 | | | | |
| | 3% | 1.62 | 1.65 | 2.34 | | | | |
| Silk | 5% | 2.04 | 2.58 | 2.74 | | | | |
| | 7% | 2.29 | 3.12 | 3.83 | | | | |
| | 3% | 0.82 | 1.08 | 1.31 | | | | |
| Cotton | 5% | 1.50 | 1.81 | 1.95 | | | | |
| | 7% | 2.04 | 2.58 | 2.74 | | | | |

3.2.5 EFFECT OF MORDANTING TECHNIQUE ON K/S OF PRINTING HENNA COLORANT IN ORIGINAL FORM

To study techniques of mordanting (pre-mordanting and simultaneous mordanting) of Alum/Tannic acid mordants and their effects on printing henna in original form, on wool, silk or cotton samples, and compared with non-mordanted samples, The K/S values were remarked as shown in Table (VI).

| Nature of | Dye conc. | K/S of samples | K/S of san Alum n | nples with nordant | K/S of samples with Tannic Acid mordant | | |
|-----------|-----------|--------------------|----------------------|-----------------------|--|------|--|
| fabric | % | without mordant | I | Ш | I | II | |
| | 3% | 1.85 | 2.28 | 1.98 | 2.71 | 2.46 | |
| Wool | 5% | 2.44 | 2.42 | 2.41 | 3.85 | 2.95 | |
| | 7% | 2.50 | 2.44 | 2.65 | 4.23 | 3.09 | |
| | 3% | 1.62 | 1.46 | 1.12 | 4.00 | 2.24 | |
| Silk | 5% | 2.04 | 2.10 | 1.46 | 4.42 | 2.65 | |
| | 7% | 2.29 | 1.92 | 2.23 | 4.71 | 3.55 | |
| | 3% | 0.82 | 0.85 | 0.94 | 1.88 | 1.66 | |
| Cotton | 5% | 1.50 | 1.69 | 1.41 | 2.65 | 2.14 | |
| | 7% | 2.04 | 1.94 | 2.37 | 2.82 | 2.96 | |

Table(VI): Comparison between using pre-mordanting and simultaneous mordanting on K/S of printed original-form

(I): K/S of the mordant was applied via padding technique.

(II): K/S of the mordant was incorporated direct in the printing paste.

On using Alum mordant: on printing wool samples, the highest K/S values was obtained at 7% concentration of henna dye irrespective of mordanted technique used. While on printing silk and cotton samples the highest K/S was obtained at 5% of henna on using pre-mordanted technique and 7% on using simultaneous technique.

On using tannic acid mordant: the highest K/S values was obtained at 7% of henna dye irrespective of the mordanted technique or fabric used.

3.2.6 COMPARISON BETWEEN USING PRE-MORDANTING AND SIMULTANEOUS MORDANTING ON K/S OF PRINTED NANO-FORM

Table (VII): Comparison between using pre-mordanting and simultaneous mordanting on K/S of printed nano-form

| Nature of | Dye conc. | K/S of samples | K/S of san Alum n | nples with nordant | K/S of samples with Tannic Acid mordant | | |
|-----------|-----------|--------------------|----------------------|-----------------------|--|------|--|
| fabric | % | without mordant | I | II | I | = | |
| | 3% | 1.98 | 2.62 | 2.00 | 3.08 | 2.45 | |
| Wool | 5% | 2.48 | 2.93 | 2.60 | 3.96 | 3.20 | |
| | 7% | 2.94 | 3.96 | 3.17 | 5.99 | 3.40 | |
| | 3% | 1.65 | 1.51 | 1.43 | 4.39 | 2.29 | |
| Silk | 5% | 2.58 | 2.32 | 1.80 | 4.76 | 2.37 | |
| | 7% | 3.12 | 3.84 | 2.24 | 4.98 | 3.57 | |
| | 3% | 1.08 | 0.96 | 1.16 | 2.05 | 1.73 | |
| Cotton | 5% | 1.81 | 1.79 | 1.77 | 2.70 | 2.54 | |
| | 7% | 2.58 | 3.38 | 2.55 | 4.38 | 3.17 | |

(I): K/S of the mordant was applied via padding technique.

(II): K/S of the mordant was incorporated direct in the printing paste.

It's clear from the data of Table (VII); On using Alum mordant: the highest K/S values was obtained at 7% concentration of henna dye irrespective of mordanted technique or fabrics used. In addition, Pre-mordanted technique acquired higher K/S values than simultaneous technique.

On using tannic acid mordant: the highest K/S values was obtained at 7% concentration of henna dye irrespective of mordanted technique or fabrics used.

3.2.7 EFFECT OF MINIATURIZATION ON FASTNESS PROPERTIES OF HENNA PRINTED SAMPLES

The color fastness properties of the natural fabrics printed with henna in presences of different mordants (Alum or Tannic acid) or without mordant were measured. Table (VIII) represents the data for the K/S values and the color fastness to washing, to rubbing, and to perspiration for deferent fabrics (Wool-Silk-Cotton) printed with henna before and after miniaturization.

Table (VIII): Color strength (K/S) and overall fastness properties of wool, silk, cotton printed with henna before and after miniaturization

a) On wool Before miniaturization

| | Samples before miniaturization | | | Washing fastness | | Rubbing fastness | | Perspiration | | | |
|-----------------|---|--------------------|-------------------|------------------|-----|---------------------|-----|--------------|-----|----------|-----|
| c id | Int | Dye conc. | K/S of | | | | | Acidic | | Alkaline | |
| Printe fabri | Image Image <t< td=""><td>gm / 100 ml H₂O</td><td>Printed fabric</td><td>Alt.</td><td>St.</td><td>Dry</td><td>Wet</td><td>Alt.</td><td>St.</td><td>Alt.</td><td>St.</td></t<> | gm / 100 ml H₂O | Printed fabric | Alt. | St. | Dry | Wet | Alt. | St. | Alt. | St. |
| | ut int | 3 | 1.85 | 4-5 | 4-5 | 4 | 4 | 3-4 | 3-4 | 4 | 4 |
| tho | tho | 5 | 2.48 | 4-5 | 4-5 | 4 | 3-4 | 3-4 | 3-4 | 4 | 4 |
| | Wi ma | 7 | 2.50 | 4-5 | 4-5 | 4 | 3 | 3-4 | 3-4 | 4 | 4 |
| - | | 3 | 2.28 | 4-5 | 4-5 | 4-5 | 4 | 3-4 | 3-4 | 4 | 4 |
| Voo | Vith Jun | 5 | 2.42 | 4-5 | 4-5 | 4-5 | 3-4 | 3-4 | 4 | 4 | 4 |
| > | > 4 | 7 | 2.44 | 4-5 | 4-5 | 4-5 | 3-4 | 3-4 | 4 | 4 | 4 |
| | د <u>ب</u> ع | 3 | 2.71 | 4-5 | 4-5 | 4 | 4 | 3-4 | 3 | 4 | 4 |
| | Vith ann acid | 5 | 3.85 | 4-5 | 4-5 | 4 | 4 | 3-4 | 3 | 4-5 | 4 |
| | > <u>"</u> " | 7 | 4.23 | 4-5 | 4-5 | 4 | 3-4 | 3-4 | 3-4 | 4-5 | 4 |

Alt. : Alteration St.: Staining

b) On wool After miniaturization

| | Samples After miniaturization | | | Washing fastness | | Rubbing | | Perspiration | | | |
|-------------------|----------------------------------|---------------------------------|-----------------------------|---|-----|---------|-------------|--------------|--------------|-------------|-----|
| Printed fabric | Mordant | Dye conc. gm / 100 ml H₂O | K/S of Printed fabric | K/S of Printed fabric Alt. St. Dry We | | Wet | Aci Alt. | dic St. | Alka Alt. | line St. | |
| | ut int | 3 | 1.98 | 4-5 | 4-5 | 4-5 | 4 | 4 | 4 | 4-5 | 4 |
| Witho morda | 5 | 2.84 | 4-5 | 4-5 | 4-5 | 4 | 4 | 4 | 4-5 | 4 | |
| | 7 | 2.94 | 4-5 | 4-5 | 4 | 3 | 4 | 4 | 4-5 | 4 | |
| _ | | 3 | 2.62 | 4-5 | 4-5 | 4-5 | 4 | 4 | 4 | 4 | 4 |
| νοσ | Vith | 5 | 2.93 | 4-5 | 4-5 | 4-5 | 3-4 | 4 | 4 | 4-5 | 4-5 |
| > | > 4 | 7 | 3.96 | 4-5 | 4-5 | 4-5 | 3-4 | 4-5 | 4 | 4-5 | 4-5 |
| | r ic | 3 | 3.08 | 4-5 | 4-5 | 4 | 3-4 | 3-4 | 3-4 | 4-5 | 4 |
| | Vit l ann acid | 5 | 3.96 | 4-5 | 4-5 | 4 | 3 | 4 | 4 | 4-5 | 4-5 |
| | 2 2 2 | 7 | 5.99 | 4-5 | 4-5 | 4 | 3-4 | 4 | 4 | 4-5 | 4-5 |

On silk:

a) Before miniaturization

| | Samples before miniaturization | | | Washing fastness | | Rubbing | | Perspiration | | | |
|-------------------|-----------------------------------|---------------------------------|-------------------|------------------|-----|----------|-----|--------------|-----|----------|-----|
| - | it | Due como | x/ct | | | lastness | | Acidic | | Alkaline | |
| Printec fabric | Mordar | gm / 100 ml H ₂ O | Printed fabric | Alt. | St. | Dry | Wet | Alt. | St. | Alt. | St. |
| | ut ant | 3 | 1.62 | 4-5 | 4-5 | 4 | 4-5 | 4 | 4 | 4 | 4 |
| tho | itho | 5 | 2.04 | 4-5 | 4-5 | 4-5 | 4-5 | 4 | 4 | 4 | 4 |
| | N N | 7 | 2.29 | 4-5 | 4-5 | 4 | 4 | 4-5 | 4 | 4 | 4 |
| | | 3 | 1.46 | 4-5 | 4-5 | 4 | 3-4 | 4 | 4 | 4 | 4 |
| Silk | lun Vit | 5 | 2.10 | 4-5 | 4-5 | 3-4 | 3 | 4 | 4 | 4 | 4 |
| | > < | 7 | 1.92 | 4-5 | 4-5 | 3 | 2-3 | 4 | 4 | 4-5 | 4-5 |
| | | 3 | 4.00 | 4-5 | 4-5 | 3-4 | 3 | 4 | 4 | 4 | 4 |
| Vith | Vith anni acid | 5 | 4.42 | 4-5 | 4-5 | 3-4 | 3 | 4 | 4 | 4 | 4 |
| | | 7 | 4.71 | 4-5 | 4-5 | 3 | 3 | 4 | 4 | 4 | 4 |

Alt. : Alteration St.: Staining

b) After miniaturization

| Samples After miniaturization | | | | Washing fastness | | Rubbing | | Perspiration | | | | |
|----------------------------------|--------|------------------------|--|-----------------------------|----------|---------|-----|--------------|--------------|------------|--------------|-------------|
| Printed | tabric | Mordant | Dye conc. gm / 100 ml H ₂ O | K/S of Printed fabric | Alt. St. | | Dry | Wet | Acio Alt. | dic St. | Alka Alt. | line St. |
| Silk | | Without mordant | 3 | 1.65 | 4-5 | 4-5 | 4-5 | 4 | 4-5 | 4 | 4-5 | 4 |
| | | | 5 | 2.58 | 4-5 | 4-5 | 4-5 | 4 | 4-5 | 4-5 | 4-5 | 4-5 |
| | | | 7 | 3.12 | 4-5 | 4-5 | 4 | 4 | 4-5 | 4-5 | 4-5 | 4-5 |
| | | Fannic With id Alum | 3 | 1.51 | 4-5 | 4-5 | 4 | 4 | 4-5 | 4-5 | 4-5 | 4 |
| | Silk | | 5 | 2.32 | 4-5 | 4-5 | 4 | 3-4 | 4-5 | 4-5 | 4-5 | 4-5 |
| | | | 7 | 3.84 | 4-5 | 4-5 | 4 | 3-4 | 4-5 | 4-5 | 4-5 | 4-5 |
| | | | 3 | 4.39 | 4-5 | 4-5 | 4 | 3-4 | 4-5 | 4-5 | 4-5 | 4-5 |
| | | | 5 | 4.76 | 4-5 | 4-5 | 4 | 3-4 | 4-5 | 4-5 | 4-5 | 4-5 |
| | | With T ac | 7 | 4.98 | 4-5 | 4-5 | 3-4 | 3-4 | 4-5 | 4-5 | 4-5 | 4-5 |

Alt. : Alteration St.: Staining

On cotton

a) Before miniaturization

| Samples before miniaturization | | | | Washing fastness | | Rubbing | | Perspiration | | | |
|-----------------------------------|---------------------|---------------------------------|-----------------------------|------------------|-----|----------|-----|--------------|-----|----------|-----|
| _ | It | Due como | K/S of Printed fabric | | | lasiness | | Acidic | | Alkaline | |
| Printec fabric | Mordar | gm / 100 ml H ₂ O | | Alt. | St. | Dry | Wet | Alt. | St. | Alt. | St. |
| | Without mordant | 3 | 0.82 | 4-5 | 4 | 4-5 | 4-5 | 4 | 3-4 | 4 | 4 |
| | | 5 | 1.50 | 4-5 | 4 | 4-5 | 4-5 | 4 | 3-4 | 4 | 4 |
| | | 7 | 2.04 | 4-5 | 4 | 4-5 | 4 | 4 | 3-4 | 4 | 4 |
| 5 | | 3 | 0.85 | 4-5 | 4-5 | 4-5 | 4-5 | 4 | 4 | 4 | 4 |
| otto | Vith Iun | 5 | 1.69 | 4-5 | 4-5 | 4-5 | 4 | 4 | 4 | 4 | 4 |
| ŭ | > < | 7 | 1.94 | 4-5 | 4-5 | 4-5 | 3-4 | 4 | 3-4 | 4 | 4 |
| | _ .2 | 3 | 1.88 | 4-5 | 4-5 | 4-5 | 4-5 | 4 | 4 | 4 | 4 |
| | Vith ann acid | 5 | 2.65 | 4-5 | 4 | 4-5 | 4 | 4 | 4 | 4 | 4 |
| | | 7 | 2.82 | 4-5 | 4 | 4-5 | 3-4 | 4 | 4 | 4-5 | 4 |

Alt. : Alteration St.: Staining

b) After miniaturization

| Samples After miniaturization | | | | Washing fastness | | Rubbing | | Perspiration | | | |
|----------------------------------|----------------------|---------------------------------|-----------------------------|------------------|-----|---------|-----|--------------|------------|--------------|--------------|
| Printed fabric | Mordant | Dye conc. gm / 100 ml H₂O | K/S of Printed fabric | Alt. St. | | Dry Wet | | Acic Alt. | lic St. | Alka Alt. | aline St. |
| | Without mordant | 3 | 1.08 | 4-5 | 4 | 4-5 | 4-5 | 4 | 4 | 4 | 4 |
| | | 5 | 1.81 | 4-5 | 4 | 4-5 | 4-5 | 4 | 4 | 4-5 | 4 |
| | | 7 | 2.58 | 4-5 | 4-5 | 4-5 | 4 | 4 | 4 | 4-5 | 4 |
| ç | With Alum | 3 | 0.96 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4 | 4-5 | 4 |
| otto | | 5 | 1.79 | 4-5 | 4-5 | 4-5 | 4 | 4-5 | 4-5 | 4-5 | 4-5 |
| 3 | | 7 | 3.38 | 4-5 | 4-5 | 4-5 | 4 | 4-5 | 4-5 | 4-5 | 4-5 |
| | _ 0 | 3 | 2.05 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 | 4-5 |
| | Vith anni acid | 5 | 2.70 | 4-5 | 4-5 | 4-5 | 4 | 4-5 | 4-5 | 4-5 | 4-5 |
| | N Ta a | 7 | 4.38 | 4-5 | 4-5 | 4-5 | 4 | 4-5 | 4-5 | 4-5 | 4-5 |

Alt. : Alteration St.: Staining

Before miniaturization. The table's data shows that washing properties ranging from very good to excellent whereas the rubbing and perspiration ranging from good to very good. While after the miniaturization: the overall properties ranging from very good to excellent.

With respect to fastness properties, there is a tendency of improvement of the perspiration and rubbing fastness while keeping the washing fastness unaltered after miniaturization of the henna dye.

4 CONCLUSIONS

• Nanoscale henna natural dye with particle size less than 100 nm were successfully prepared by using ultrasonic starrier.

- Increasing the dye concentration from 3 to 7gm/100ml water increases the average of dye particle size from 28.1 nm to 102.9 nm at nano-form, and from 103.7nm to 377.5nm at original form. With greater tendency of the nanoparticles to aggregate / agglomerate.
- Irrespective of the fabric used, the K/S values of nano-henna samples are higher than original-henna samples.
- Nano-henna in presence or absence of mordant acquired higher K/S values than original-henna.
- Nano-henna dye could be used successfully instead of the original dye incorporated with Alum mordant when applied on silk and cotton fabrics.
- On using tannic acid mordant: the highest K/S values was obtained at 7% concentration of henna dye irrespective of mordanted technique or fabrics used or dye particles size used.
- Washing fastness properties are not significantly affected by miniaturization but rubbing and perspiration properties have a better values on using nano-henna than original henna.

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