

**New technique using light for fixing solubilized vat dyes****Assist. Prof. Dr/ Amr Emad El-Dien Allam****Assitant.Prof of Textile Dyeing and Finishing Technology –Faculty of Applied Arts,  
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University, Egypt.****Abstract:**

In this study the researcher tries to work out a new technique using light for fixing solubilized vat dyes, Innovations that have increased recently for creating smart textiles, That interact with surrounding environmental conditions like photochromism , Thermochromism and Chemochromism, However the dyes that are affected by these environmental conditions here are called Anthrasol Blue IBC solubilized vat dyes .

**Keywords:** solubilized reduced vat dyes, photochromism, developed by light.

**نبذة مختصرة**

في هذه الدراسة حاول الباحث اكتشاف طريقة جديدة باستخدام الضوء لتثبيت صبغات الأحواض الذائبة، لقد زادت مؤخرا ابتكارات من أجل تصنيع المنسوجات الذكية التي تتفاعل مع ظروف البيئة المحيطة مثل الضوء والحرارة والتفاعلات الكيميائية، في هذا البحث الصبغات التي تتأثر بهذه الظروف البيئية المحيطة تسمى صبغات الأحواض الذائبة أنثرازول زرقاء.

**كلمات مفتاحية:** solubilized الأصباغ ذات القيمة المضافة أقل ، فوتوكروم ، وضعت بال ضوء.

**مشكلة البحث**

من الغير مناسب ان يتم إختزال صبغة الأحواض لإذابتها ومن الغير عملي أيضا بيعها في الصورة المختزلة لأنها سوف تتأكسد في الهواء الجوي عائدة إلى صورتها الغير ذائبة. الحل لهذه المشكلة هو ان يقوموا المصنعون بتحويل الصورة الذائبة الحامضية إلى الإستر منها مثل تفاعل الصورة الحامضية مع حمض الكبريتيك. عند التعرض للهواء هذه الصورة الذائبة من الصبغة تتحول للصورة الغير ذائبة الملونة من الصبغة، علي عكس الصبغات القديمة عندما كانت تتحول الصبغة من الشكل الأصلي إلى اللون المطلوب في حمام الصباغة من خلال عملية أكسدة.

**هدف البحث**

في هذه الدراسة يفضل الباحث استخدام صبغات الأحواض قبل الإختزال . في هذا البحث يتم التركيز علي توليد عملية تلوين مختلفة للألياف السيلولوزية مثل الأقمشة القطنية. الهدف هو إمداد تسهيلات للتصاميم الفردية تقابل احتياجات المستهلكين وإرضائها.

**أهمية البحث**

إستخدام طريقة جديدة لتثبيت صبغات الأحواض الذائبة وعودتها مرة اخري إلى الإستخدام بطرق فنية حديثة.

**منهجية البحث**

بحث تجريبي تحليلي.

**أهم النتائج التي تم التوصل إليها**

بعد التجارب ودراسة العوامل المختلفة من ( درجة حرارة حمام الصباغة، تركيز الصبغة، تركيز الملح، تركيز نترات الصوديوم، وقت الصباغة، زمن التعرض لضوء الشمس وزمن التعرض للأشعة فوق بنفسجية) تم إثبات أن أفضل ظروف للصباغة هي في درجة حرارة 70 مئوية لمدة 45 دقيقة في حمام صباغة يحتوي علي ( 5 جرام صبغة، 5 جرام نترات الصوديوم، 5 جرام ملح ) في 100 مل ماء وعينة وزنها 2 جرام ثم التعرض لضوء الشمس لمدة 20 دقيقة.

**Introduction**

Vat dyes are important dyes for dyeing and printing on cotton and cellulosic fibers .They have excellent all round fastness, which includes perspiration and rubbing fastness [4]. The soluble vat dye applied in practical procedure of dyeing on cotton fabric but it difference between insoluble vat dyes and soluble vat dyes that instead of oxygen free bath, it developed by sunlight [13].

**Photochromism**

When discussing the term photochromic, one should first clear out the term “chromic “. chromism is a process that induces changes on the molecular level like cleavage of the chemical bonds or changes on the level of molecular conformations [6]. In most of the cases chromism is based on changes occurring inside of the molecule, among electrons. this especially implies „ Pi " and „ d" electron positions so that the phenomenon is induced by various outside stimulus bearing the ability of altering electronic density of the compound or a substance [3] . chromism refers to the phenomenon in which color is the result of abroad spectral interactions among incident light and material [9].These interactions may be categorized into following five groups :

- Reversible change in color.
- Absorption and reflection of light.
- Absorption of energy followed by the emission of light .
- Absorption of light and energy transfer or transformation of energy.
- Use manipulation of light [1].

**These chromic effects are caused by :**

- Per cyclic reactions.
- Cis-trans isomerization.
- Intermolecular group transfers.
- Intermolecular hydrogen transfers.
- Dissociation (cleavage of the bonds).
- Electron transfer [12].

Phenomenon including a change in color of a chemical compound is named according to external stimulus causing the reaction, either physically or chemically. Many but not all of these reactions are reversible [10].

## Soluble vat dyes

It's inconvenient to have to reduce your vat dyes in order to dissolve them. However, it is impractical to sell the reduced form of the dye, because it will oxidize in the air, back to the insoluble form [5]. The solution to this problem nowadays is for the manufacturer to convert the soluble leuco acid form of the dye to the leuco ester, such as by reacting the leuco acid with sulfuric acid. The leuco form of this solubilized dye can be regenerated by removing the ester group chemically [13]. On exposure to air this soluble leuco form of the dye gets converted to colored insoluble form of the dye [1]. Unlike the past dyes when the dye was done in the original form and convert the color in the dye bath to its original color with oxidation process, However, The powder form of the solubilized vat dyes are stable to storage, if properly stored away from sunlight and air (moisture, oxygen and carbon dioxide). Their solutions are also stable if properly stored if they come in contact with acidic fumes or oxygen a part of the dye is wasted [7].

In such work, the researcher would get back to using vat dyes before reduction. This piece of work focus on regenerating such process for coloring different cellulosic materials including cotton fabrics. The aim is to provide individual designs facilitates everyday consumer needs.

## EXPERIMENTAL

### MATERIALS:

Fabric: the fabric used for the work was finer counts of 100% bleached, mercerized cotton fabric. The fabric structure of used cotton is plain weave. It produced from GIZA Company [8].

### Dyestuffs and Chemicals:

Anthrasol Blue IBC, solubilized vat dye, Dystar Company, Singapore, Azo dye used photosensitizers, Sodium Carbonate  $\text{Na}_2\text{CO}_3$ , Poly vinyl alcohol (P.V.A), Sodium nitrite  $\text{NaNO}_2$ , Zinc Formaldehyde Sulfoxalate, Sulfuric acid  $\text{H}_2\text{SO}_4$  [9,11.]

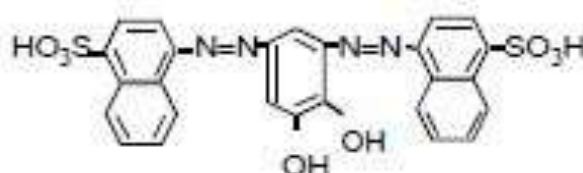
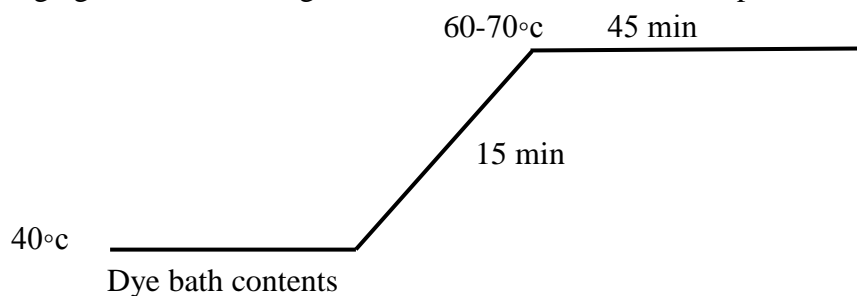


Fig (1) Chemical structure of Azo dye [14]

## Dyeing process

Immerse the cotton fabric (2gm weight of the sample) in the dye bath containing (100 ml water, 5gm sodium carbonate, 5gm soluble vat dye and 5gm nitrite sodium) raise the temperature to 60 -70 °c for 45 min, extract the sample from the dye then immerse it in Azo and P.V.A liquor ratio (1:10) solution to make the dye developed by sunlight and get dark shades, let it in presence of direct sunlight for 20 min.

Washing off: Dyed samples were rinsed with zinc formaldehyde sulfoxalate (5g/L) then immerse in sulfuric acid (10%) for 1 min to get dark shades , soaped with a solution containing 5g/l nonionic detergent for 1 min, after wash the samples left it air dried.



This thesis studying the effect of dyeing temperature, dyeing time, dyeing concentration, Sod. Carbonate and Sod. Nitrite concentration. Furthermore studying, exposing time to sunlight and ultraviolet light.

## Color measurements and Fastness Properties

### Color measurements

A Perkin Elmer spectrophotometer was used to measure the K/S values, Model Lambda35 equipped with integrated sphere according to Kubelka-Munk equation .

$$K/S = (1-R)^2 / 2R$$

### Where:

- R: Decimal fraction of the reflectance of dyed samples
- K: Absorption coefficient
- S: scattering coefficient [2].

### Fastness Properties

1. The fastness to crocking was measured according to the test method 8-2013.
2. The fastness to perspiration was measured according to the test method 15-2013.
3. The fastness to light was measured according to the test method 16-2011.
4. The fastness to laundering was measured according to the test method 61-2013.
5. The pH value was measured according to the test method 81-2012.
6. The fastness to water was measured according to the test method 106-2013.
7. The fastness to sea water was measured according to the test method 107-2013.

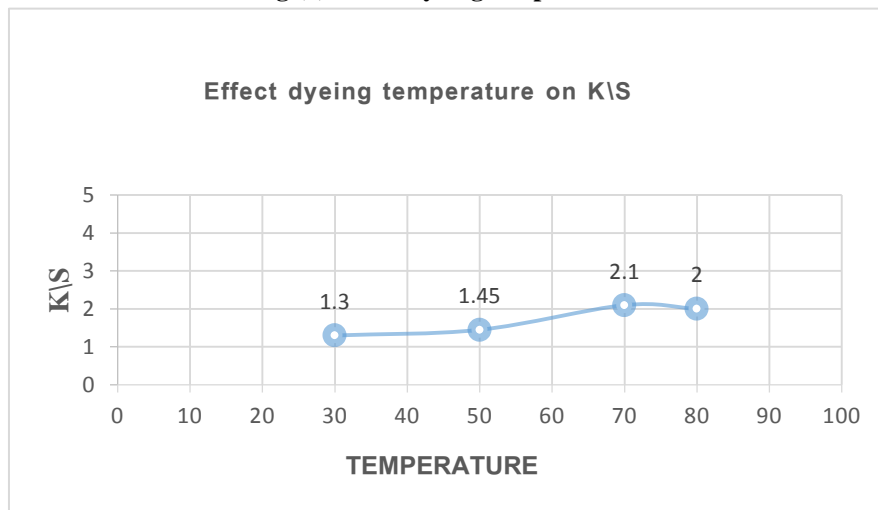
The samples were prepared in standard atmosphere for 24 hours before performing tests: Temperature ( $20 \pm 2$  ° C) and Humidity % ( $65 \pm 2$  %) according to the test method 8-2013.

## Results and Discussion

### Color measurements

#### 1. Effect dyeing temperature on K/S:

Fig (2) Effect dyeing temperature on K/S

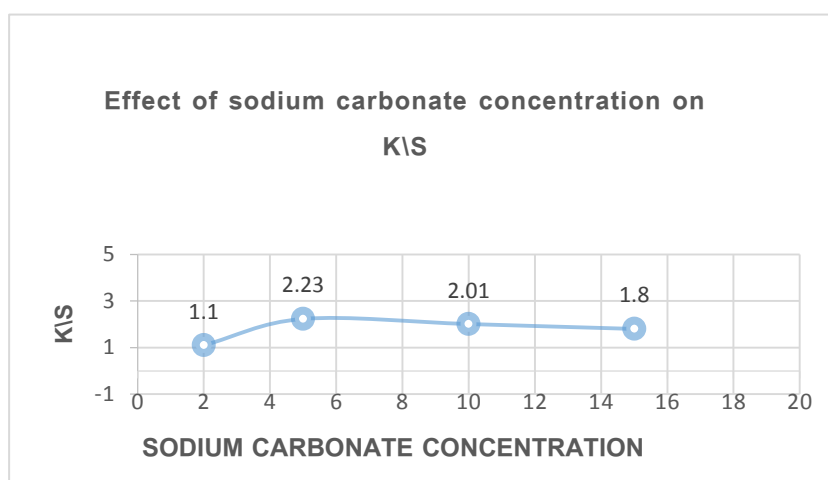


Dyeing bath contents: 5gm Anthrasol Blue IBC, 5gm Na<sub>2</sub>Co<sub>3</sub>, 5gm Na No<sub>2</sub>, dyeing temp., X (30, 50, 70, 80°C), time 45 min. exposing 20 min to sunlight.

**Figure (2)** shows the effect of dyeing Temp. on color strength (K/S), the result revealed that a higher K/S values for the sample dyed at 70°C, and the lowest value for the samples were for those dyed at 30°C. According to the results shown above low Temp. affects the produced results negatively due to the dye pamphlet that recommended to keep the Temp. between 60-70°C.

#### 2. Effect of sodium carbonate concentration on K/S:

Fig (3) Effect of sodium carbonate concentration on K/S



Dyeing bath contents: 5gm Anthrasol Blue IBC, sodium carbonate concentration, X (2, 5, 10, 15gm), 5gm Na No<sub>2</sub>, dyeing temp. 70°C, time 45 min. exposing 20 min to sunlight.

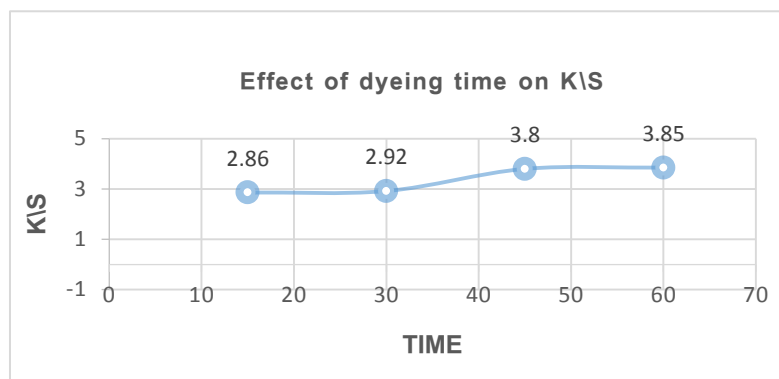
**Fig (3)** shows the color strength (K/S), results which revealed that the higher K/S values for the sample dyed with 5gm salt, was better than higher concentrations of salt .

When the concentration of sodium carbonate was 30gm and 50gm the samples failed because after immersed in Azo:P.V.A solution it made like a plastic film coating the fiber.

It is obvious that increasing the salt affects the results which could be due to the alkaline medium of the dye bath, it should not be so high and also it reacts with the polyvinyl alcohol turns into plastic film.

### 3. Effect of dyeing time on K/S :

**Fig (4) Effect of dyeing time on K/S**



**Dyeing bath contents: 5gm Anthrasol Blue IBC, 5gm Na<sub>2</sub>CO<sub>3</sub>, 5gm Na NO<sub>2</sub>, dyeing temp.70°C, Time of dyeing, X (15, 30, 45, 60 min),exposing 20 min to sunlight.**

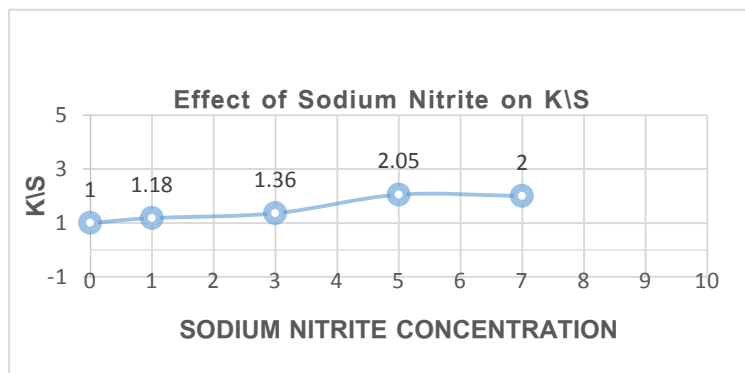
Results obtained and shown in Fig (4), shows that samples dyed for 15 and 30 min shows similar results ,but the best results was at 45 min.

Best results were obtained while increasing the time, probably such results could be due to the reaction was sufficient with the fiber in 45 min.

Accordingly 45 min would be an optimum time for dyeing .

### 4. Effect of sodium nitrite on K/S:

**Fig (5) Effect of sodium nitrite on K/S**

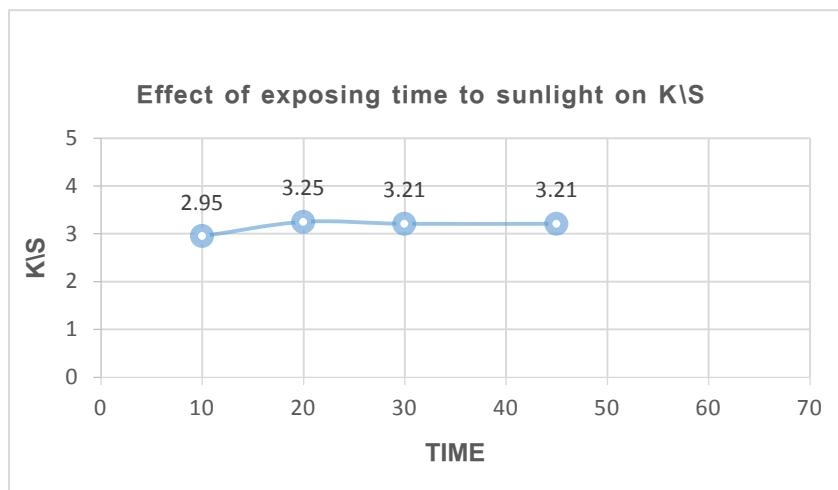


**Dyeing bath contents: 5gm Anthrasol Blue IBC, 5gm Na<sub>2</sub>CO<sub>3</sub>, Concentration of sodium nitrite, X (1, 3, 5,7gm), dyeing temp.70°C, Time of dyeing, 45 min, exposing 20 min to sunlight.**

**Fig (5)** shows the color strength (K\S), the results revealed that were a higher K\S values for the sample dyed with 5gm of nitrite sodium was the best .  
Increasing the amount of sodium nitrite could affect the process badly this may be due to making the dye molecules less attached with the fiber.

**5. Effect of exposing time to sunlight on K\S :**

**Fig (6) Effect of exposing time to sunlight on K\S**

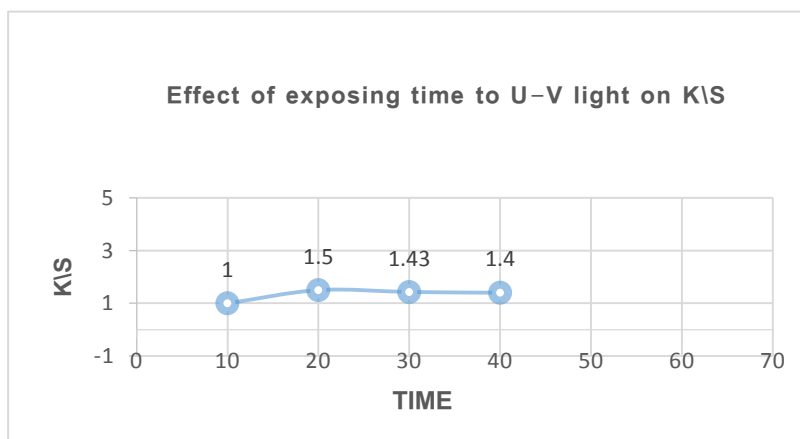


**Dyeing bath contents: 5gm Anthrasol Blue IBC, 5gm Na<sub>2</sub>Co<sub>3</sub>, 5gm NaNo<sub>2</sub>, dyeing temp.70°c, Time of dyeing, 45 min, exposing time to sunlight, X (10, 20, 30, 45 min).**

**Fig (6)** shows the color strength (K\S), results revealed that a higher K\S values for the sample exposed to sunlight for 20 min.  
Best results were obtained at 20 min due to the effect of sunlight on polyvinyl alcohol, when it get drier the reaction stopped as in 30, 45 min.

**6. Effect of exposing time to Ultra Violet light on K\S:**

**Fig (7) Effect of exposing time to Ultra Violet light on K\S**



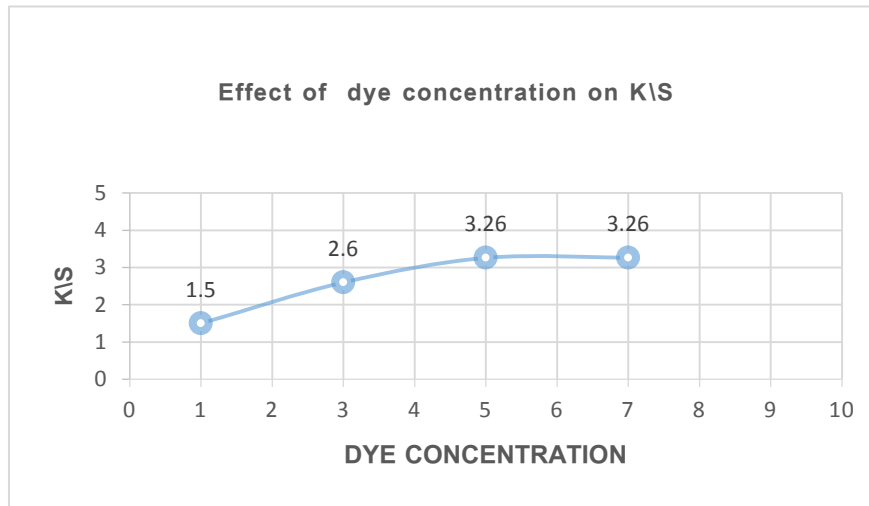
**Dyeing bath contents: 5gm Anthrasol Blue IBC, 5gm Na<sub>2</sub>Co<sub>3</sub>, 5gm NaNo<sub>2</sub>, dyeing temp.70°c, Time of dyeing, 45 min, exposing Time to Ultra Violet light, X (10, 20, 30, 40 min).**

**Fig (7)** shows the color strength (K\S), results revealed that a higher K\S values for the sample exposed to U-V light for 20 min.

Best results were obtained at 20 min due to the effect of Ultra Violet light on polyvinyl alcohol, when it get drier the reaction stopped as in 30, 40 min.

### 7. Effect of dye concentration on K\S:

**Fig (8) Effect of dye concentration on K\S**



**Dyeing bath contents: Anthrasol Blue IBC dye concentration, X (1, 3, 5, 7gm), 5gm Na<sub>2</sub>CO<sub>3</sub>, 5gm NaNO<sub>2</sub>, dyeing temp.70°C, Time of dyeing, 45 min, exposing 20 min to sunlight.**

**Fig (8)** shows the color strength (k\S), results revealed that a higher K\S values for the sample with 5gm dye in 100 ml water.

Best results were obtained at 5gm dye due to this amount was sufficient to 2gm of the fabric and 100 ml water, for 7gm the dye will be redundant for the liquor ratio for the fabric.

From the previous results show the best dye bath with the Anthrasol Blue IBC dye were 5gm of dye in 100 ml water for 2gm weight of the sample, 5gm of sodium carbonate, 5gm nitrite sodium for 45 min at temperature 70°C and exposing to sunlight for 20 min after been treated with Azo: P.V.A solution, rinse with zinc formaldehyde sulfoxalate then sulfuric acid (10%) for 1 min to convert the dye to get dark shades, soaping with a solution containing 5g/l nonionic detergent for 15 at 40°C min, after wash the samples left it air dried.



**Fastness properties of dyed cotton Sample with Anthrasol Blue IBC solubilized vat dye**

The results shown in Table (1, 2) express the fastness properties of the dyed cotton Sample with Anthrasol Blue IBC solubilized vat dye in sunlight for 20 min.

Samples	laundrying 40°C	Perspiration Acidic	Water	Seawater
<b>Color change</b>	<b>4</b>	<b>4.5</b>	<b>4.5</b>	<b>4.5</b>
<b>Staining on:</b>				
<b>Wool</b>	<b>4.5</b>	<b>4.5</b>	<b>4.5</b>	<b>4.5</b>
<b>Viscose</b>	<b>5</b>	<b>4.5</b>	<b>5</b>	<b>4.5</b>
<b>Silk</b>	<b>4.5</b>	<b>4.5</b>	<b>4.5</b>	<b>4.5</b>
<b>Nylon</b>	<b>3.5</b>	<b>5</b>	<b>4.5</b>	<b>5</b>
<b>Cotton</b>	<b>3.5</b>	<b>4.5</b>	<b>5</b>	<b>4.5</b>
<b>Acetate</b>	<b>5</b>	<b>5</b>	<b>5</b>	<b>5</b>
<b>Self-staining</b>	<b>4.5</b>	<b>N/A</b>	<b>N/A</b>	<b>N/A</b>

Table (1): Fastness properties of the dyed cotton Sample with Anthrasol Blue IBC solubilized vat dye in sunlight for 20 min.

Samples	PH	Crocking		Light
		Dry	Wet	Color change : 4
	<b>6.75</b>	<b>4.5</b>	<b>4</b>	

Table (2) shows the color fastness properties of the dyed cotton Sample (Crocking – PH)

**Which N/A: Not Applicable**

According to Table (1, 2) the results show such a good performance, the laundering fastness were as good as grade 4 in color change .also the staining on the multi-fiber was between 3.5 and 5 and the self-staining 4.5 . The acidic perspiration shows an excellent performance to staining between 4.5 and 5 with respect to color change that shows a grade 4.5. Also the fastness to water and sea water shows a great performance in color change and staining according to Table (1) grades between 4.5 and 5. According to Table (2) the color change in fastness to light gave a very good grade 4, and for the crocking the dry sample shows a 4.5 grade and the wet sample grade 4 and this such an excellent result due to the vat dyes. The pH value of the solution was extracted from the sample 6.75 and this a convenient result for using the dye in any field.

## Conclusion

The performance of Anthrasol Blue IBC (solubilized vat dyes) was successfully achieved , the dye was fixed by sunlight ,on industrial purpose the dyed samples have a very good impact on fastness properties ( laundering ,water ,seawater ,perspiration ,crocking , light and pH value ) and have an artistic view to get light and dark shades , From these results it was clearly that solubilized vat dyes has a good impact on the functional properties of the fabric. Extra work is needed in the same direction to improve vat dyes in order to make them more competitive in such work, we used Anthrasol Blue IBC dye it is important for such dye to back to life, since we used other different dyes for such work and did not success .and manufactures no longer use this dye in its original form, they modified it to suit their production. But we proved that the original form we use in this study has various uses artistic or industrial.

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