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Abstract

In recent years, the textile industry proceeds towards creation of new technologies to reduce the energy and water consumption. The use of ultrasonic in textile wet processing is one way to achieve that purpose. The present work investigates the effects of ultrasonic energy on dyeing of silk fabrics with Chlorophyll as a natural dye. Silk fabrics were dyed with Chlorophyll dye in an ultrasonic bath by the conventional exhaustion method. The parameters that affect the dyeing process, such as dye concentration, dye bath pH, salt concentration, and temperature, time and power energy ultrasonic values have been explored. Concentration of tannic acid was also studied as a mordant in the dyeing process. The results show that the color strength as well as fastness properties of the dyed silk fabrics were enhanced on using the ultrasonic technique compared with the conventional method in presence of 5% tannic acid.

Keywords: Ultrasonic energy; Silk fabrics; Chlorophyll natural dye; Tannic acid; Dyeing; fastness properties.

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Introduction

In recent decades ultrasonic has established an important position in different industrial processes and has started to revolutionize environmental protection. This technique has been studied and used for a variety of applications in liquids, dispersions and polymers.

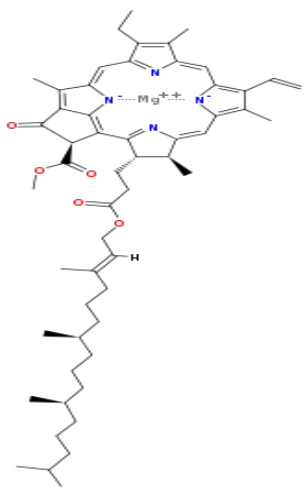
In the textile field ultrasonic technique was applied in the mechanical processes (weaving, finishing and making-up for cutting and welding woven, non woven and knitted fabrics) and wet processes (sizing, scouring, bleaching, dyeing, etc.) [1-8].

On the other hand, natural dyes have been used for coloring and printing fabrics to protect the environment from the indiscriminate exploitation and pollution by industries. Recently, the potentiality of using natural dyes in textile coloration as UV-protection and antimicrobial has been investigated [9-11]. Moreover, several studies on the application of natural dyes have been repotted [12-19].

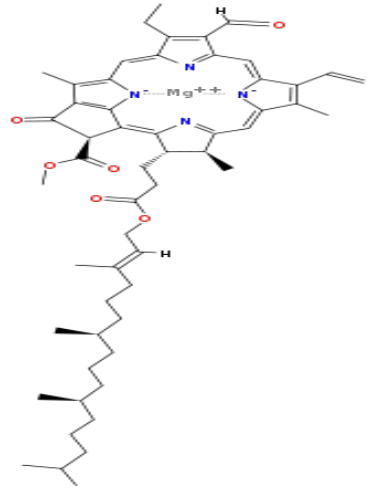
Chlorophyll is a green compound found in leaves and green stems of plants. Due to the green color of chlorophyll, it has many uses as dyes and pigments. It is used in coloring soaps, oils, waxes and confectionary. The first application of chlorophyll derivatives for dyeing wool, acetate fibers and cotton were reported [21, 22].

Anciently, it was assumed that chlorophyll was a single compound but in 1864 it was showed by the spectroscopic analysis that chlorophyll was a mixture of two compounds, chlorophyll-*a* and chlorophyll-b represented in Figure (1).

The problem of using the natural pigment, chlorophyll, as ecologically pure dye for textile fibers was discussed [20]. That problem is related to the low exhaustion of the dyes and to the fastness of the dyed fabrics. Attempts to overcome these problems have been mainly focused on the use of metallic salts as mordents, which are traditionally used to improve fastness properties or exhaustion and to develop different shades with the same dye [23-25].



Structure of Chlorophyll (a)



Structure of Chlorophyll (b)

(Fig.1)

There were many studies interested with dyeing of the natural fibers with natural dyes using ultrasonic technique [26-28]. As continuity, the present work aims to study the dyeing of silk fabrics with Chlorophyll as a natural dye using ultrasonic energy to save temperature and time as well as to protect the environment. Factors affecting the dyeability and fastness properties were studied. The results obtained were compared with the conventional exhaustion dyeing method. The effect of using tannic acid as a mordant on the dyeing process was investigated.

Experimental

Materials

Fabrics

Silk fabrics of plain weave (29/19) in both weft and warp supplied from Akhmim Upper Egypt. The fabrics were washed in an aqueous bath containing 2g/l soap at pH 9 and 45^{0}C for 30 min. The fabrics were thoroughly washed with warm water then with cold water, squeezed and air dried at ambient temperature.

Dyestuffs

Chlorophyll-100-WSP: A commercial product by HANSEN`S Co. based on Chlorophyll complex 95% and maltoodextren.

Chemicals and auxiliaries:

Sodium sulphate, acetic acid and Tannic acid are laboratory grade chemicals. Non ionic detergent (Hostapal CV.ET, Hoechest).

Apparatus

Thermostated CREST benchtop 575 HT ultrasonic cleaner of capacity 5.75 L, frequency 38.5 kHz and with a maximum 500 Watt output was used. The output power levels are from 100 up 500 Watt.

Dyeing methods:

Dyeing silk fabrics using ultrasonic method and conventional method:

Silk fabrics (1g) was dyed with Chlorophyll 100-WSP (1-5%), using sodium sulphate (zero to 11g/l) at L.R 1:50 and the pH was adjusted to (4-7) using acetic acid. The samples were added to the dye bath at temperature varied from (30-70^{0}C) for(5-80 min.), the power energy of ultrasonic was varied (100 to 500 watt) then the dyed fabrics washed in an aqueous bath containing 3 g/l of non ionic detergent at 50^{0}C for 30 minutes then rinsed and dried.

Testing methods:

Color measurements*:*

The dyed samples were subjected to color measurement using reflection spectrophotometer model Perkin-Elmer Lambda 3B UV/V. Relative color strengths (K/S values) were determined using the Kubelka- Munk equation [29] .

$$K/S = \frac{\left(1-R^2\right)}{2R} - \frac{\left(1-R_0^2\right)}{2R_0}$$

where, R is Decimal fraction of the reflectance of the dyed sample, R_0 is Decimal

fraction of the reflectance of the undyed sample, K is Absorption coefficient, and S is Scattering coefficient.

Fastness properties:

Fastness properties of dyed samples were tested according to ISO standard methods. The color fastness to rubbing of the dyed fabrics was tested according to S 105-X12(1987), the color fastness to washing was tested according to ISO 105-C02 (1989), and ISO 105-E04 was used to test the color fastness to perspiration. The dyed samples were subjected to tests, for fastness to light by AATCC test method 16-1993.

Results and Discussion:

Ultrasonic vibrations travel in the form of a wave similar to the way of light travels. However, unlike the light waves, which can travel in a vacuum, the ultrasonic requires an elastic medium such as a liquid or a solid for its transmission. The frequency range normally employed in ultrasonic non-destructive testing and thickness gauging is 100 kHz to 50 MHz. Although the ultrasonic behaves in a manner similar to the audible sound, it has a much shorter wavelength. This means it can be reflected off very small surfaces such as defects inside materials. [30] The occurrence of cavities depends upon several factors such as the frequency and intensity of waves, temperature and vapor pressure of liquids. The present work make use of the advantages of these waves for dyeing silk fabrics with Chlorophyll as a chlorinated pigment.[31]

Factors affecting dyeing properties:

Effect of dye concentration:

The effect of dye concentration on the color strength of dyed silk fabrics with chlorophyll, expressed as K/S, using ultrasonic and exhaustion methods were cited in Figure 2.

It is noticed that the K/S increases till 4% in case of ultrasonic method then decreases while in case of conventional method the K/S increases as the dye conc. increases to 5%. There are many hypotheses to explain the possible action of ultrasonic on the dyeing system. This action can be explained in the following three fold effects:

i) Dispersion: breaking up of micelles and high molecular weight aggregates into uniform dispersions in the dye bath.

ii) Degassing: expulsion of dissolved or entrapped gas or air molecules from fiber capillaries and interstices at the crossover of fabric into liquid and removal by cavitations, thus facilitating dye-fiber contact.

iii) Diffusion: accelerating the rate of diffusion of the dye inside the fiber by piercing the insulating layer covering the fiber and accelerating the interaction or chemical reaction, between dye and fiber.

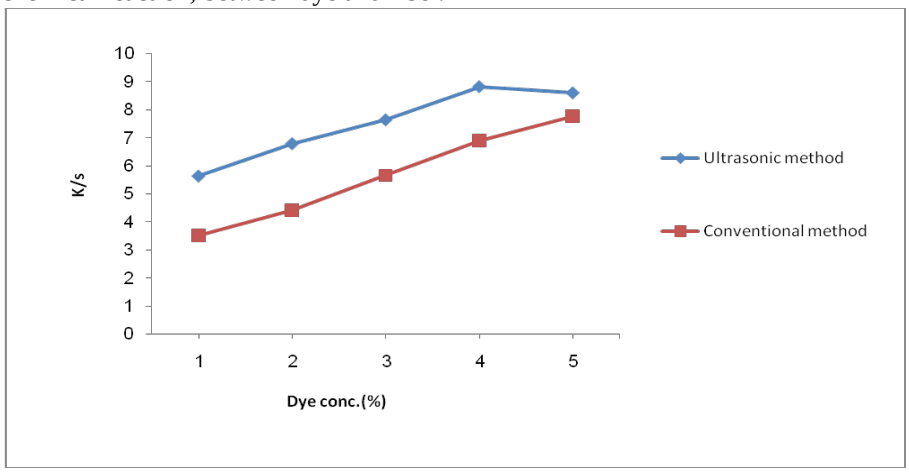


Fig (2): The effect of dye concentration on K/S of dyed silk fabrics with Chlorophyll using ultrasonic and conventional exhaustion methods.

Dyeing condition with the ultrasonic method: L.R, 1:50; pH, 6.5; salt conc., 1g/l; time, 30 min; ultrasonic energy, 400 W; temp., 50 ^{O}C.

Effect of pH values:

The effect of pH values were obtained to the dyed silk fabrics with Chlorophyll using ultrasonic and exhaustion method .The results were illustrated in fig.(3)where we can notice that the best color strength were obtained at pH 5 in case both of ultrasonic and exhaustion method .The ultrasonic results were higher than the conventional one.

These results depended on the structure of the dye and the dye technique .The structure of Chlorophyll, in which the acetyl groups were hydrolyzed into carboxylic groups, gives an idea about the suggestion attraction between dye and fabric which will depend on Van dar waal forces beside the hydrogen bond and the possibility of ionic bond which seemed increased in acidic medium.

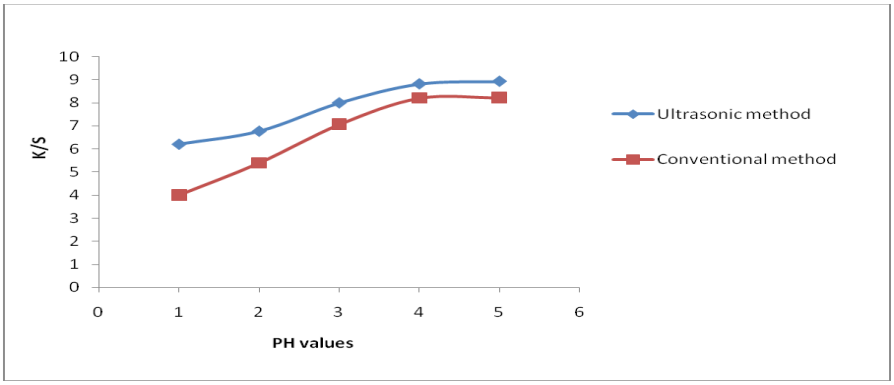


Fig.(3) : Effect of pH values on the color strength(K/S) of dyed silk fabrics with Chlorophyll using ultrasonic and conventional methods.

Dyeing condition:

Ultrasonic method: L.R 1:50, 4%dye, 1g/l salt, 30min., 400 W and 50^{0}C.

The difference in the results between two methods was due to the technique of dyeing where the influence of ultrasonic on the dyeing system is suggested to have three fold effects:

i) Dispersion: breaking up of micelles and high molecular weight aggregates into

Uniform dispersions in the dye bath.

ii) Degassing: expulsion of dissolved or entrapped gas or air molecules from fiber capillaries and interstices at the crossover of fabric into liquid and removal by cavitations, thus facilitating dye-fiber contact.

iii) Diffusion: accelerating the rate of diffusion of the dye inside the fiber by piercing the insulating layer covering the fiber and accelerating the interaction or chemical reaction, between dye and fiber.

Effect of salt concentration:

Figure 4 shows the K/S of dyed silk fabrics with chlorophyll using ultrasonic and exhaustion dyeing method in presence of different concentrations of sodium sulphate (zero-11g/l). It is clear that the best color strength was obtained in case of ultrasonic method in the absence of the salt whereas 1 g/l of this salt was necessary

to give the best results in case of the exhaustion method reflecting the advantage of the ultrasonic technique.

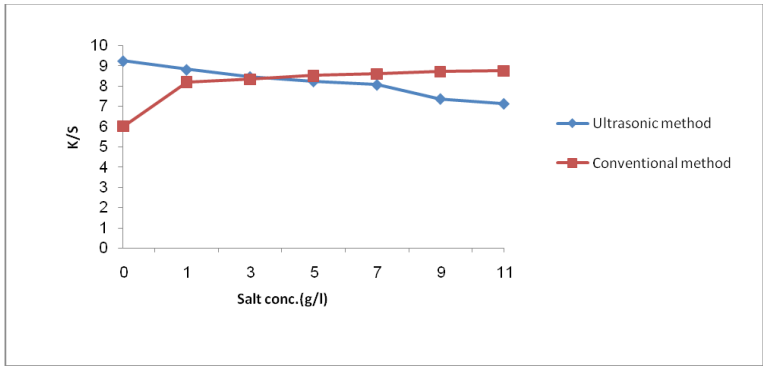


Figure (4): Effect of salt concentration on K/S of dyed silk fabrics with Chlorophyll using ultrasonic and exhaustion methods.

Dyeing condition of ultrasonic method: L.R, 1:50; dye conc. 4%; pH 5; time, 30min.; ultrasonic energy, 400 W; and temp., 50 ^{0}C.

Effect of dyeing temperature:

Figure 5 showed the effect of temperature on the dyeability of silk fabrics with Chlorophyll dye using ultrasonic compared with exhaustion method. It is obvious that the color strength, in case of ultrasonic technique, increases with increasing the dyeing temperature until 60^{0}C then decreases whereas in case of the exhaustion method the K/s increases with increasing the dyeing temperature within the range studied. Moreover, the ultrasonic technique results in higher K/S extents than that of the exhaustion method. This could be attributed to ultrasonic power, at ambient temperatures, which provides a de-aggregation effect on the dye molecules leading to improvement in dye diffusion and better dyeability than conventional method.

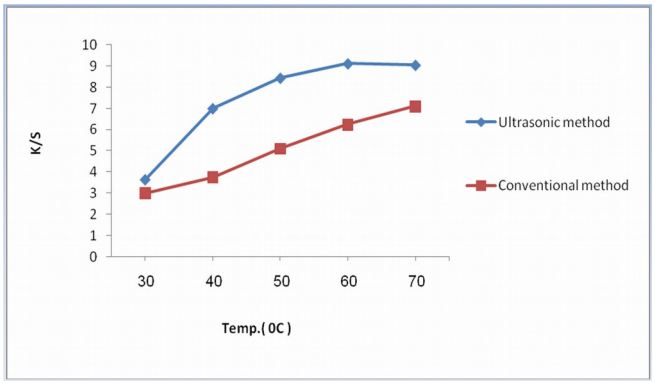


Figure (5): Effect of dyeing temperature on the color strength (K/S) of dyed silk fabrics with Chlorophyll using ultrasonic and exhaustion methods.

Dyeing condition of ultrasonic method: L.R, 1:50; dye conc. 4%; pH, 5; time, 30min.; ultrasonic energy, 400 W.

Effect of dyeing time:

Figure 6 shows the effect of the variation in the dyeing time on dyeability of dyed silk fabrics with ultrasonic technique compared with that of exhaustion method.

It is clear that, for a given set of conditions, the K/S values of dyed fabrics in any case of ultrasonic or conventional methods, increase as the time increased till 60 min then decrease which may be attributed to a partially hydrolysis taking place for the dye fixed on the dyed fabrics surfaces [27].

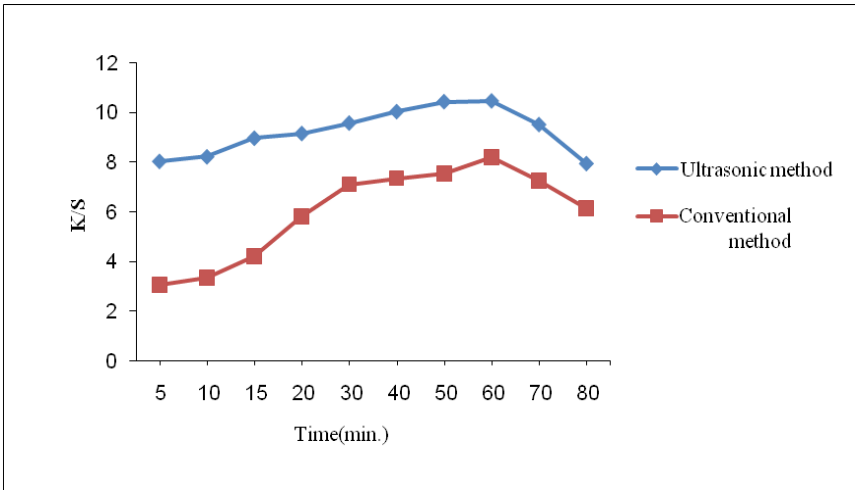


Figure (6): Effect of dyeing time on the color strength (K/S) of dyed silk fabrics with Chlorophyll using ultrasonic and exhaustion methods.

Dyeing condition of ultrasonic method: L.R, 1:50; dye conc. 4%; pH, 5; ultrasonic energy, 400 W; temp., 60^0C.

Effect of ultrasonic power:

The effect of ultrasonic power on the color strength of dyed silk fabric with Chlorophyll using different power values (100-500 watt) was represented in Figure

7. The results show that the color strength increase as the ultrasonic power increases. The best color strength was obtained at 500 watt confirming the effect of ultrasonic power on the dyeing process.

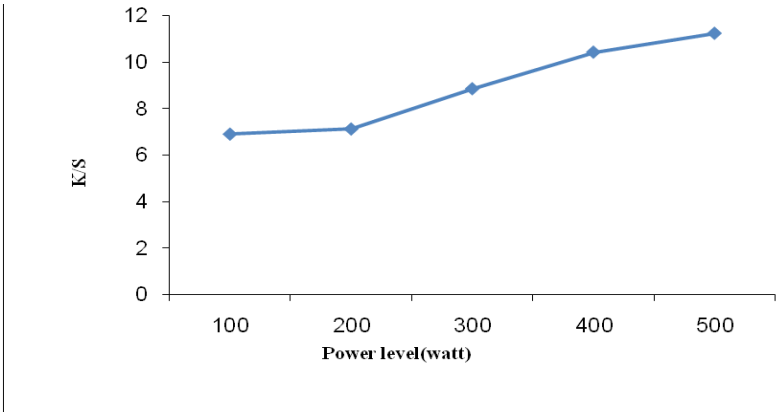


Figure (7): Effect of power level on the color strength (K/S) of dyed silk fabrics with Chlorophyll using ultrasonic and conventional method.

Dyeing condition of ultrasonic method: L.R, 1:50; dye conc. 4%; pH, 5; time, 60 min.; temp., 60^0C; ultrasonic energy, 500 W.

Effect of using Tannic acid as mordant at different types of mordanting:

Under the previous optimum conditions, silk fabrics was dyed using the ultrasonic or conventional method in presence of different concentrations of tannic acid as a mordant. Table 2 illustrates the effect of tannic acid on k/s of the dyed fabrics. The results obtained clarify that best color strength were obtained at 5% tannic acid for both dyeing methods.

Table (2): Effect of tannic acid conc. on the color strength (K/S) of dyed silk fabric using ultrasonic or conventional exhaustion methods.

Tannic acid conc. (%)	Ultrasonic method (K/S)	Conventional method (K/S)
0	10.4	8.1
1	10.89	8.67
3	11.72	8.98
5	12.74	9.2

7	11.34	8.63
9	11.22	8.43

Dyeing condition of u**ltrasonic method**: L.R, 1:50; dye conc., 4%; pH, 5; time, 50 min.; temp., 60^0C.

Tannic acid of concentration 5%, as a mordant, was added to the dyeing bath at different stages of dyeing, pre-, simultaneous and post-dyeing, to find out the best suitable stage to add the mordant. The results obtained from Figure 8 show that the mordanting with tannic acid at the pre-dyeing stage is the best.

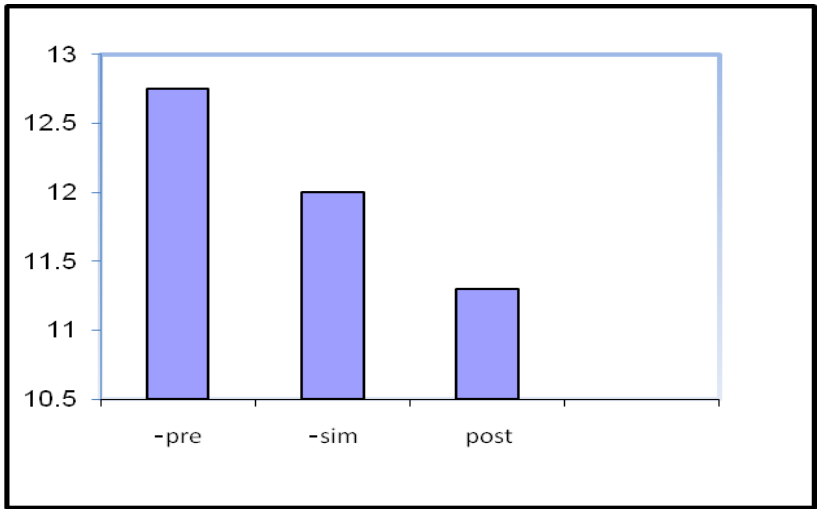


Figure (8): The effect of tannic acid on the color strength of dyed silk fabric with Chlorophyll using ultrasonic method at different stages of dyeing, pre-, simultaneous and post-dyeing.

Fastness properties:

Fastness properties of dyed silk fabrics with Chlorophyll using ultrasonic, conventional exhaustion methods and mordant samples by using premordanting method were carried out, the results were cited in Table1.The results indicated that the samples gives fair to good for both dyeing methods while by using mordant gave an improvement in the washing fastness. The fastness properties obtained using mordant is excellent.

The color fastness to washing, rubbing, perspiration and light, in case of pre mordant colored fabrics ranges from very good to excellent. The overall fastness properties of colored fabrics are very good.

Table 3: Fastness properties of dyed and mordant silk fabrics with Chlorophyll using ultrasonic and conventional exhaustion methods

Dyeing methods	Washing			Perspiration						Rubbing		Light fastness
				Acidic			Alkaline					
	Alt.	St*	St**	Alt	St*	St**	Alt	St*	St**	Dry	Wet	
conventional exhaustion method	3:4	3:4	3:4	3:4	3:4	3:4	3	3	3	3	3	6
conventional exhaustion method ***premordant***	4	4	4	4	4	4	3:4	3:4	3:4	3:4	3:4	7
Ultrasonic ***method***	4	4	4	4	4	4	3:4	3:4	3:4	3:4	3:4	7
Ultrasonic ***method premordant***	4:5	4:5	5	5	5	5	5	5	5	5	5	7

St.* = Staining on cotton
St. **= Staining on wool
Alt . = Alternation in color

Dyeing condition of u**ltrasonic method**: L.R, 1:50; dye conc., 4%; pH, 6; time, 50 min.; temp., 60^0C; tannic acid conc., 5%.

Conclusions:

Silk fabrics were dyed with Chlorophyll natural dye by using ultrasonic energy. The results of factors affecting dyeability on the color strength were carried out and compared with the results of conventional exhaustion method .It was found that the use of ultrasonic energy in dyeing processes offers advantages from the point of view conservation of energy and time. The salient features of ultrasonic dyeing in this work can be summarized as follows:

- Low Temperature Dyeing at 60°C (savings of energy 40 %)
- Increased Exhaustion & Fixation (increased by 20%)
- Reduced Dyeing Time 50 min. (reduced by16 %)
- Uniform dyeing.

Using tannic acid as mordant improved the color strength and the fastness properties

So, the results of investigation offer a new viable method for dyeing of silk fabrics by environmentally friendly method and saving energy.

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ملخص البحث باللغة العربية

إستخدام الموجات الصوفية لصباغة الاقمشة الحريرية بطريقة أمنة بيئيا

يتناول البحث صباغة الاقمشة الحريرية بأستخدام صبغة طبيعية هى صبغة الكلوروفيل المستخلصة من الاوراق النباتية الخضراء بأستخدام الموجات الفوق صوتية بهدف المحافظة على البيئة و توفير الطاقة و الوقت.

عند استخدام طريقة الموجات الفوق صوتية فى الصباغة و مقارنتها بطريقة الصباغة بالاستنفاذ تم الحصول على قوة لون للاقمشة المصبوغة افضل من الصباغة بالاستنفاذ مع توفير فى درجة الحرارة و الوقت حيث امكن الصباغة عند 60 درجة مئوية لمدة 50 دقيقة بدون استخدام ملح كبريتات الصوديوم. و قد امكن تحسين قوة اللون و خواص الثبات باستخدام حمض التانيك بتركيز 5% كمثبت طبيعى امن بيئيا.