Microwave Assisted Dyeing of Cellulose with Direct Dyes

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Abstract

In this research work, dyeing of cotton fabric with direct dyes carried out with exhaustion method by microwave system. The effect of microwave power, time of dyeing, electrolyte concentration, dye concentration and L:G was outlined. The results of preliminary experiments on the effect of microwave on the dye solution and the fabric itself clearly showed that the microwave process has no effect on the cotton fabric and direct dyes. Other results indicated that an increase in the microwave power causes an increase in the dye absorption by the fabric. However the time of dyeing process could also be very effective on the dye absorption. Therefore, extension of dyeing time up to 20 min causes higher exhaustion but further increase in the time of dyeing leads to a decrease in the dyeing absorption. Presence of electrolyte as an absorption ionic material in dyeing process, leads to increase the exhaustion. It can be concluded that the effect of electrolyte and dye concentration in the microwave heating system were similar to the normal exhaustion dyeing. The effects of liquor to good ratio in microwave irradiation were not the same as the normal exhaustion dyeing. Because, the exhaustion increases with increasing of L:G to 100:1 and then decreases in higher L:G. Finally the results of washing fastness showed a small improvement in the washing fastness on the fabric dyed by microwave irradiation.

Keywords: Dyeing, Microwave, Direct Dyes, Cotton, Exhaustion.

Introduction

For many years dielectric heating has been used commercially as a method of heating materials that are poor conductors of heat, e.g. wood and plastics. The material is exposed to a high frequency electromagnetic field and rapid internal heating is caused by molecular friction or agitation, induced by rapid reversals of the polarity of the electric field.

The term dielectric heating is generally applied to the use of high frequency equipment operating with working frequencies below 80 MHz in legally prescribed frequency bands. The conversion of electrical–field energy into heat increases in proportion to the working frequency and to the square of the strength of the electrical field. However, there are only limited possibilities of increasing the field energy by increasing the frequency (i.e. by a factor of 3 from 13.6 to 40.68 MHz). Increasing the field strength is not practical since this often results in electrical breakdown in and around the material being treated, with consequent arcing, sparking or flashovers damaging the material [1].

Microwave has wavelength between about 1m and 1mm corresponding to frequency from 300 MHz up to 300 GHz. Microwave heating, a form of electric heating, is understood as the generation of heat in materials of low electrical conductivity by the action of high- frequency electric field [2]. It must be stressed, however that microwave heating should be regarded as a form of dielectric heating, since the physical principles are similar [1].

Under the influence of an alternating field, the dipole molecules undergo oscillations in response to the high frequency field polarity changes. The intermolecular friction produces high-frequency energy which is first absorbed then transformed into thermal energy. Substances with a symmetrical molecular structure, such as benzene, cannot be heated in a high-frequency field, since they lack the necessary dipole characteristics. Apart from the dipoles molecules, freely movable ions can also be influenced by an alternating electric field. The moves to and from in response to the high- frequency polarity changes, and thereby collide with each other. In the course of these actions, high-frequency energy is again absorbed and transformed into thermal energy. Generally, this proportion of transformed energy–especially at the almost exclusively employed frequency of 2450MHz is relatively small compared to the proportion produced by dipole oscillations. However, electrolytes, salt solutions, glass and ceramics are an exception at high temperatures. As for these substances, a considerable amount of heat increases upon a reduction of the frequency, this may be attributable to ion movement [2]. Also these principles can be illustrated by the simple ammonia molecule. Ammonia has a dipole moment because of its asymmetric configuration and two stable positions. Rapid reversal of polarity in an electric field results in constant alteration of the two stable positions which is accompanied by development of heat. The average time of inversion is approximately 2.5×10^{-13}sec. Nevertheless, distinctions should be made between dielectric and microwave heating. Thus, what is normally referred to as dielectric heating involves a lower frequency and as high a field strength as possible, without electrical breakdown of the field, whereas microwave heating uses a much higher frequency and a comparatively low field strength. At this point it should be stated that most textile fibers are not regarded as 'Lossy' materials. A lossy material is one in which the overall molecular structure, or a group within the structure, can be induced to resonate at frequencies similar to the frequency of the microwave radiation, resulting in a dielectric loss of the input energy which is converted into heat within the material. Since fibers are themselves comparatively non-lossy, water or another lossy substance must be present. The microwave energy heats the water, which in turns heats the fiber. Therefore moisture is required to facilitate diffusion of the dye and as a transfer medium for heat. However, it should be realized that the heating of water occurs uniformly throughout the fiber and does not involve conducting mechanism as in steaming where the heat is applied from outside the fibers [1].

The material is exposed to a high frequency electromagnetic field (e.g. 2450 MHz) rapid internal heating is caused by molecular friction, induced by rapid reversal of the polarity of the electrical field. This cause resonated under similar frequencies of microwave which produced heat [1]. The power of material for absorption of radiation depends on electrical properties and magnetic parameter of molecules [2-5]. Application of microwave as a source of thermal energy is very important and nowadays it is in favor for the textile producers. In finishing and dyeing processes of textile usually thermal energy needed and in normal heating system the transfer of heat takes place from outside of the fabric to inside of it. In this way of heating the fabric surface is warmer. This causes over drying and dye migration [6-7]. Since the microwave heating system produced heat inside the fabric, then the mechanism of heat transfer differs from thermal conductivity in normal heating system. Therefore the microwave heating system has no problem of different temperature between inside and outside of the fabric [8]. Example of application of microwave heating is a jig machine equipped with microwave's generator. The finishing by this machine showed an increase in efficiency of finishing with a decrease in the time of processing without any decrease in the fabric quality. By application of this thermal energy, faults such as uneven dyeing between first and last and also differences in edges of the fabric which normally existed in conventional method can be decreased [9]. Polli and Edoardo recommended use of a tunnel equipped with a microwave generator for drying and removing of organic waste from insects adhere to cotton. They showed that the usage of this method can improve the process ability of cotton fiber and increase the efficiency of spinning [10]. In addition, the use of microwave heating system in alkaline hydrolysis treatment of polyester (relation to normal method) can increase the hydrolysis efficiency, weight loss, wicking ability and dye uptake by polyester fabrics [11]. Also microwave heating system used for eradication of insects from wool textiles. In this method, 3min radiation of microwave may killed (100%) the egg, larval and adults stages of moth, with the lowest effect on the physical and chemical properties of wool fabric [12]. Badrosamaa and Amirshahi showed that, no changes occurred in the absorption of reactive dyes on the cotton fabric treated with microwave radiation (600 Watt) before dyeing. They also observed that usage of the microwave heating system in dyeing of cotton with reactive dyes by exhaustion method reduces the time of dyeing without any changes in dye-uptake. They showed that the microwave heating system used for absorption and fixation steps of reactive dyes on the padded cotton fabrics increases the efficiency of dyeing in shorter time in compare with the cold pad batch method [13]. There is no report on the dyeing of cotton fabric with direct dyes by microwave in exhaustion method. Thus this research work tried to use the microwave heating system for dyeing of cotton fabric with direct dyes by exhaustion method. To do this, the effects of different factor such as microwave power, time of dyeing, electrolyte concentration, dye concentration and liquor to goods ratio (L:G) was investigated.

Experimental

Materials

The fabric used, was bleached woven cotton with 120 g/m² of weight. The fabric samples dyed with direct dyes including Solophenyl Scarlet BNL 200%, Solophenyl Blue TLE, Solophenyl Yellow ARL 154% along with analytical grade of Na_2SO_4 (Merck) as electrolyte.

Methods

The exhaustion determined by Perkin Elmer-Lambda 25 Spectrophotometer and X-RITE Reflectance Spectrophotometer. Dyeing process carried out with LG Microwave MC-2003 TRS (900 watt-power and 2450 MHz-frequency). The fabric samples strength was measured by Instron (model 4202).

Dyeing was carried out by exhaustion method with two different heating systems. In the normal heating system, cotton samples were added to the dye bath at 40ºC and then the temperature increases to boil during 20 min. The dyeing was carried out at boil for 45min and then the samples rinsed and air dried. In order to dye the samples with microwave irradiation heating system by exhaustion method, the glass container was used. Dyeing was carried out at different time of 5, 10, 15, 20, 25 and 30 min and also various microwave power including 180, 360, 540, 720 and 900 watt. Effects of electrolyte, dye concentration and L:G was determined on selected 720 watt for 15 min because of optimum results obtained in this conditions.

In order to determine the equation of absorbance via concentration, the absorption- concentration curve was drawn. Six different concentrations of dye was prepared and the absorption of each ones at λ max was measured by absorbance spectrophotometer.

The exhaustion percent was calculated by following equation:

$$\text{Exhaustion \%} = (A-B/A)*100$$

Which A is the concentration of dye solution before dyeing and B is the concentration of dye solution after dyeing.

Also by using the Kubelka-Munk equation, the K/S value was calculated.

$$K/S = (1- R)^2/2R \qquad \text{(Kubelka–Munk equation)}$$

The colour differences (ΔE) were measured by color coordinates (CIELAB D65 /10):

$$\Delta E = [(a_1*-a_2*)^2 + (b_1*-b_2*)^2 + (l_1*-l_2*)^2]^{1/2}$$

The fabric whiteness was reported according to Hunter equation:

$$W\ (\text{Hunter}) = 100 - [(100- L)^2 + 10b^2]^{1/2}$$

The fabric shrinkage percent also measured by following equation:

$$\text{Shrinkage \%} = (A-B/A)*100$$

Which A is the original distance between reference marks and B is the final (after exposure) distance between references marks.

The washing fastness of selected samples was measured according to ISO-R-105 and change of it was evaluated with gray scale.

Results and discussion

1- Effect of microwave on the dye and cotton fabric

To examine the effect of microwave irradiation on dye solution, the dye solution exposed to the microwave irradiation and absorbance spectrum of solution before and after irradiation was recorded. It can be seen from figures

1-6 that the absorbance spectrum of aliquot dye bath, before and after microwave irradiation, have no changes and the λ_{max} and color of dye bath is similar. Also, to examine the influence of the microwave irradiation on the cotton fabric, the fabric sample impregnated in the aqueous solution and subjected to the microwave irradiation and then the strength and fabric whiteness was measured. The results of whiteness and strength of cotton samples before and after microwave irradiation also showed no changes. This means the chemical and physical properties of the cotton fabric has not been changed by the microwave irradiation (table 1-2). Therefore the thermal energy produced by the microwave irradiation can be used for dyeing of cotton fabric with direct dyes.

2- Effect of dyeing time and power of microwave irradiation

The results of exhaustion against time of dyeing at different power of microwave irradiation were shown in figures 7-9 (for Solophenyl Scarlet BNL 200%). The results showed that, extension of dyeing time to 20min at a given power of microwave irradiation (180-900 watt), leads to increase the absorption of dye in comparison with normal exhaustion. Increasing of dyeing time to 25-30min with irradiation of microwave at 540, 720 and 900watt also leads to decrease the dye absorption. However, the results of dye absorption showed that an increase to the time of dyeing causes the dye absorption to increase at 180 and 360 watt.

The heat transfer was done by conduction for dyeing process in normal exhaustion method, it means that the surface of the material was first heated and then the heat transfer through the sample. Therefore, this mechanism required time for transferring the heat from the dye-bath into the fabric. While the dyeing process by microwave irradiation is differed as the mechanism of heat transfer is different. In the microwave irradiation all of the material was heated simultaneously and there is no need for transferring of heat slope from surface to inside of the material. In this way, the dye bath reaches to a specific temperature during dyeing process very soon. The results in Figures 7-9 showed that in given time of dyeing (20 min), with increasing of power of microwave, the dye absorption increases. In fact, with increasing of power of microwave, absorption of energy increases by loss material (loss material: absorptive electromagnetic such as water, electrolyte …) exist in the aqueous media. Therefore increasing of kinetic energy of the dye molecule leads to decrease the time required for transferring of dye molecule into the fiber. In addition, at this conditions, the rate of swelling of cotton fiber was increased. Thus, the system was reached to equilibrium rapidly. With increasing the time of exposure of microwave irradiation (increasing of dyeing time to 25-30) and bringing in mind this reality that the linkage of the dye to the fabric is weak and can be break down at high temperature. Then number of dye can be separated from the fabric and migrated to the dye-bath, as a result the exhaustion decreases (the amount of migration related to the size and structure of the dye). At low power (such as 180 watt) the energy of system was low, and then an increase to the time of dyeing leads to increase of exhaustion.

3- Effect of electrolyte

Effect of electrolyte in dyeing of cotton with direct dyes in normal exhaustion method is the neutralization of fiber surface charge and decreasing of electrical repulsion between the dye and fiber. This leads to increase the absorption of dye by goods and uniformity of dyeing. The results in figure 10 showed that changes of K/S against concentration of electrolyte in cotton dyeing with direct dyes by exhaustion method with microwave heating is similar to the normal heating as the presence and increase of concentration of electrolyte (0-5 g/l), caused an increase to the K/S. Also it can be observed that the dyeing by microwave irradiation is more depending upon the presence and increase of electrolyte in the dye-bath. The reason is in addition to the influence of electrolyte in the normal exhaustion dyeing, the microwave radiation can be also absorbed by ions [1, 4, 6 and 7].

4- Effect of concentration of dye

The results of changes of K/S of cotton fabric dyed with direct dyes (normal and microwave exhaustion method) against concentration of dye showed in figure 11. It cab seen that, in normal heating dyeing with increasing of dye concentration, the exhaustion increases. This process was also done for dyeing of cotton with some dyes (at 0.25-1% concentration) by microwave irradiation. The results indicated that a little sensitivity to dye concentration exist which is based on the structure of dye, specially with electrical charged dyes. These could absorbed electromagnetic wave directly, same as water and electrolyte and vibrated with frequency equal to the frequency of microwave. In this way, the dye molecules in addition to losing absorbed energy to form heating energy they could produce electrical layer around them which may increase the chance of effective contact between the dye and fiber (i.e. increasing exhaustion).

5- Effect of L:G

In dyeing of cotton fabric with direct dyes by normal exhaustion method, depending on the kind of machine, different L:G may used. However it is desirable to use a low L:G process also at low L:G seems to have a lower electrical repulsion between dye and fiber and can be obtain an increased exhaustion. The results showed that with increase of L: G from 40:1 to 100:1 the K/S increases, higher increase to 150:1 decreases the K/S of the samples dyed with microwave irradiation (figure 12). It can be concluded that the energy of microwave irradiation can cover certain volume of an aqueous solution. It can be also resulted that at L:G of 40:1 with microwave irradiation the complete energy can not be discharged. With increasing of volume of aqueous solution the energy of irradiation produced can be discharged and then it can be increased the number of dye molecules with higher energy. This could be arised the chance of contact between dye molecules and fiber which increases the exhaustion. There was no free microwave radiation with increase of volume of solution and therefore distribution of microwave energy between high numbers of particle was encountered. Therefore, in this condition each particle has a lower energy and at a given time of dyeing causes a reduction in exhaustion.

6- Evaluation of washing fastness

The results of washing fastness showed in table 3. The results indicated that the washing fastness of samples dyed with microwave irradiation a little improved and staining on the white cotton and wool fabrics reduced.

Conclusions:

The results of this research showed that microwave irradiation with the conditions used in this experiment has no influence on the structure of direct dyes and also cotton fabrics. Therefore the thermal energy produced by the microwave irradiation can be used for dyeing process. In comparison of cotton dyeing with direct dyes by normal condition and microwave irradiation, it was observed that the microwave irradiation, reduces the time of dyeing with increasing in the rate of dyeing. This may increase the efficiency. Meanwhile with the microwave irradiation the limitation of selection of L:G isn't the same as normal heating, and also the microwave irradiation causes a relatively improvement (0.5-1 gray scale unit) in the washing fastness.

Tables and figures

Table 1: component of dye and degree of whiteness in CIELAB system for samples before and after microwave treatment

Samples	L*	a*	b*	C*	h	Whiteness
without treatment	91.70	-0.09	3.91	3.91	91.35	85.11
20´ (180w)	88.68	-0.17	3.41	3.41	93.17	84.94
20´ (900w)	90.52	-0.07	4.15	4.15	90.98	83.81

Table 2: Strength and elongation up to rapture of cotton samples before and after microwave treatment

Samples	White sample*	20´ (180w)	20´ (900w)
Force (kgf)	80.3	87.9	87.2
Elongation (mm)	13.7	23	24
Rate: 125 mm/min distance between two:20cm			

Table 3: Washing fastness and staining of samples dyed with two method normal and microwave

Samples dyed	C.I.DIRECT RED 98		C.I.DIRECT BLUE94		C.I.DIRECT YELLOW 106	
	Microwave	Normal	Microwave	Normal	Micro wave	Normal
Washing fastness	4	3	3	3	3/4	2/3
Staining of cotton	3/4	3	4	3/4	3/4	3
Staining of wool	4/5	4/5	4/5	4/5	4/5	4

*: sample without treatment.

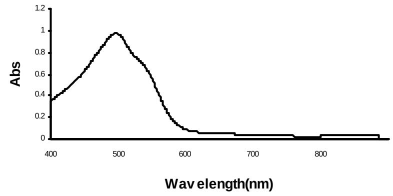

Figure2: Absorbance spectrum of aqueous solution of dye before treated with Microwave.

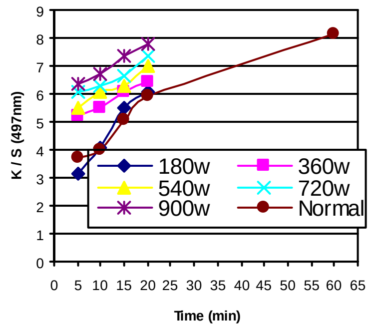

Figure 3: K/S vs. dyeing times for cotton fabric that dyeing with direct dye by normal and microwave exhaustion (dyeing process is done at different power of microwave and time of 5-20min)

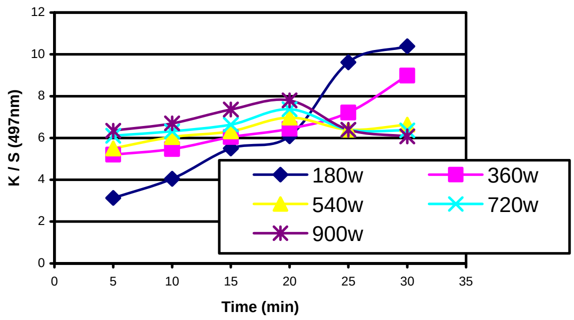

Figure 4: K/S vs. dyeing times for cotton fabric that dyeing with direct dye (dyeing process is done in different power of microwave at 5 to 30 min)

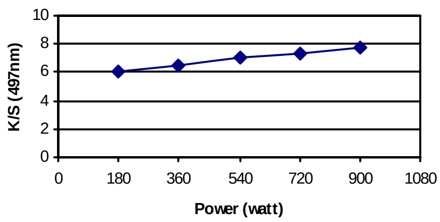

Figure5: K/S vs. power of microwave in cotton dyeing with direct dye at 20 min.

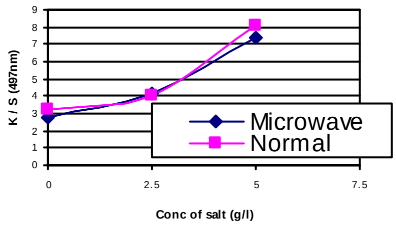

Figure6: K/S vs. concentration of salt for dyeing of cotton fabric with direct dye by two methods exhaustion (normal and microwave).

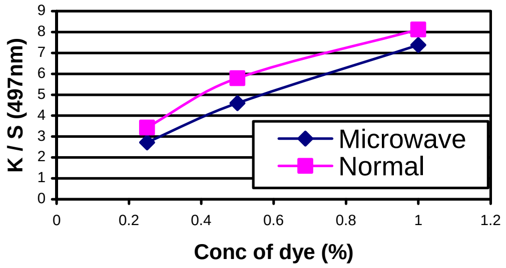

Figure 7: K/S vs. concentration of dye for dyeing of cotton fabric with direct dye by two methods exhaustion (normal and microwave).

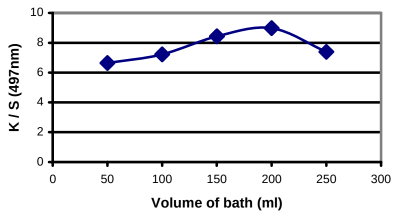

Figure 8: K/S vs. L:G in dyeing of cotton fabric with direct dye by microwave heating.

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