Egypt. J. Chem. **Vol. 64**, No. 11 pp. 6591 - 6598 (2021)

Egyptian Journal of Chemistry

http://ejchem.journals.ekb.eg/

Comparison between Low and High Temperature Dyeing of Polyester Fabrics Dyed with Disperse Dyes derived from Enaminones

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Abstract

Some disperse dyes were prepared and applied in dyeing polyester fabrics in an attempt to compare dyeing at low temperatures and high temperatures by studying the dye uptake. Also, the optimal dispersing agent ratio for both methods was studied, which ranged from 0.5 to 2%. . Finally, the fastness properties were evaluated for two dyeing methods represented by light fastness, washing fastness, rubbing fastness and perspiration fastness, where very good results were obtained, except for light fastness, the results were satisfactory.

Keywords: Low temperature dyeing, disperse dyes, high temperature dyeing, dyebath reuse.

1. Introduction

Since the discovery of synthetic fabrics, the use of disperse dyes has been continuously increased in textiles using simple exhausting techniques [1-8]. One method of dye bath reuse is to reconstitute the dye bath by adding the required amount of dyes and chemicals to prevent and reduce contamination. Dye bath reuse has long been recognized as a strategy to prevent pollution and reduction of cost [9-12]. It is known that when using low temperature dyeing of PET fibers, carriers are used for improvement adsorption and acceleration of the dispersal of dyes dispersed in the fibers. However, most of the carriers are toxic to humans and aquatic organisms and during dyeing and rinsing, a large amount of carriers are released into the wastewater which negatively affects and harms the environment. In contrast, when high temperatures are used to dye PET fibers, these carriers are not used for improvement adsorption and acceleration of the dispersion of dyes dispersed in the fibers, where the temperature at 130 °C and the subsequent high pressure play a role in obtaining a greater color depth if the two methods are compared. Also, the dyeing bath is almost free of dye residues, thus reducing the remaining substances in the wastewater, which affects positively on the environment [13-21]. In this study, polyester fibers were dyed with separate dyes in two different ways, namely, dyeing at low temperatures and high temperatures, and comparing them to find out the best.

2. Materials and Methods

The disperse dyes were prepared according to the method that we published in our previous research and checked by mass spectroscopy, essential examination, FT-IR, and ^{1}H-NMR spectroscopy [1].

Study the optimum concentration of dispersing agent.

The disperse dyes 1-6, a dispersion of the dyes were produced by dissolution of the appropriate amount of dyes (3% shades) in 2 ml DMF and then added drop wise with stirring to the dye bath (liquor ration 1:30) containing different concentration (0.5, 1, 1.5, 2%) of levegal MDL as anionic dispersing agent (TANATEX chemicals). The pH of the dye bath was adjusted to 5.5 with aqueous acetic acid, and the wetted-out polyester fabrics were added. We performed dyeing by raising the dye bath temperature to 130°C at a rate of 3°C/min and holding it at this temperature for 60 min. After they were cooled to 50°C, the dyed fibers were rinsed with cold water and reduction-cleared (1 g/L sodium hydroxide, 1 g/L sodium hydrosulfite, 10 min, 80°C). The samples

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Receive Date: 21 August 2021, Revise Date: 30 August 2021, Accept Date: 03 September 2021
DOI: 10.21608/ejchem.2021.91909.4357

were rinsed with hot and cold water and, finally, air-dried.

Dyeing procedure

A- First dyeing

The disperse dyes 1-6, a dispersion of the dyes were produced by dissolution of the appropriate amount of dyes (3% shades) in 2 ml DMF and then added drop wise with stirring to the dye bath (liquor ration 1:30) containing (1.5%) of levegal MDL as anionic dispersing agent (TANATEX chemicals) and TANAVOL EP 2007 (1%) as anionic eco-friendly carrier (TANATEX chemical) in case of dyeing at 100 °C or just use dispersing agent in case of dyeing at 130°C. The pH of the dye bath was adjusted to 5.5, and the wetted-out polyester fabrics were added. We performed dyeing by raising the dye bath temperature to 100 or 130°C at a rate of 3°C/min and holding it at this temperature for 60 min. After they were cooled to 50°C, the dyed fibers were rinsed with cold water and reduction-cleared (1 g/L sodium hydroxide, 1 g/L sodium hydrosulfite, 10 min, 80°C). The samples were rinsed with hot and cold water and, finally, air-dried.

B- Dyebath reuse

After dyeing, the dyebath was analyzed and reconstituted with the necessary amount of fresh water to maintain a constant liquor ratio of the original volume. Residual dyebath pH was measured in order to keep pH at 5.5. Dyebath reuse in dyeing was being carried out by the same procedures of the previously two techniques. Finally a reduction-cleared using sodium hydroxide (3 g/L) and sodium hydrosulphite (2 g/L) and soaped with 2% nonionic detergent (pH 8) at 50 °C for 15 minutes to improve washing fastness.

Color Measurements

The colorimetric parameters of the dyed polyester fabrics were determined on a reflectance spectrophotometer. The color yields of the dyed samples were determined by using the light reflectance technique performed on an UltraScan PRO D65 UV/VIS Spectrophotometer. The color strengths, expressed as K/S values, were determined by applying the Kubelka-Mink equation [9].

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

Where, R is the reflectance of colored samples and K and S are the absorption and scattering coefficients, respectively.

Color fastness to washing

The color fastness to washing was determined according to the ISO 105-C02:1989 method [10]. The composite specimens were sewed between two pieces of bleached cotton and wool fabrics, and then immersed into an aqueous solution containing 5 g/L of nonionic detergents at a liquor ratio of 1:50. The bath was thermostatically adjusted to 60 °C for 30 min. After the desired time, samples were removed, rinsed twice with occasional hand squeezing, and then dried. Evaluation of the wash fastness was established using the grey scale for color change.

Color fastness to rubbing

Color fastness to rubbing was determined according to the ISO 105-X12:1987 test method. The test is designed for determining the degree of color that may transfer from the surface of the colored fabrics to another surface by rubbing. The current test can be carried out on dry and wet fabrics.

Dry crocking test

The test specimen was placed flat on the base of the crockmeter. A white testing cloth was mounted. The covered finger was lowered onto the test specimen and caused to slide back and forth 20 times. The white test sample was then removed for evaluation using the grey scale for staining.

Wet crocking test

The white test sample was thoroughly (65%) wetted with water. The procedure was run as before. The white test samples were air dried before evaluation.

Color fastness to perspiration

Two artificial perspiration solutions (acidic and alkaline) were prepared as follows according to the ISO 105-E04:1989 test method. The acidic solution was prepared by dissolving L-histidine monohydrochloride monohydrate (0.5 g), sodium chloride (5 g), and sodium dihydrogen orthophosphate dihydrate (2.2 g) in one liter of distilled water. Then, the pH was finally adjusted to 5.5 using 0.1 N NaOH. To prepare the alkaline solution, L-histidine monohydrochloride monohydrate (0.5 g), sodium chloride (5 g), and disodium hydrogen orthophosphate dihydrate (2.5 g) were all dissolved in one litter of distilled water. The pH was adjusted to 8 using 0.1 N NaOH. The fastness test was performed as follows. The 5 cm × 4 cm colored specimen was sewn between two pieces of uncolored specimens to form a composite specimen. The composite samples were immersed for 15-30 min in both solutions with well agitation and squeezing to ensure complete wetting. The test specimens were placed between two plates of glass or plastic under a force of about 4-5 kg. The plates containing the composite specimens were then held vertically in an oven at 37 ± 2 °C for 4 h. The effect on the color of the tested specimens was expressed and defined by reference to the grey scale for color change.

Color fastness to light

The light fastness test was carried out in accordance with the ISO 105-B02:1988 test method, using a carbon arc lamp and continuous light for 35 h. The effect on the color of the tested samples was recorded by reference to the blue scale for color change**.**

3. Results and Discussion

New disperse dyes 1-6 synthesized previously by us [10](Figure 1) were utilized in dyeing polyester fabrics at low temperature of 100 °C and high temperature of 130 °C to try to compare these two methods and study their stability properties.

Effect of dispersing agent on* color strength *K/S

The polyester fabrics were dyed with the disperse dyes 1-6, using the 1% carrier and at a temperature of 100 °C, we studied the use of the dispersing agent at different concentrations from 0.5-2% to study the optimum concentration giving the best value of K/S. The results set out in Table 1 indicate that the K/S values of the polyester fabrics dyed with the disperse dyes of the dyes 1, 3, 5 and 6 increase with the increase of the dispersing agent concentration and reach their highest values at a concentration of 1.5 %.

The K/S values for the polyester fabric dyed with the disperse dyes of the 2 and 4 dyes also increase with increasing dispersing agent concentration and reach their highest values at a concentration of 2%. For the high temperature dyeing, the results set out in Table 1 indicate that the K/S values of the polyester fabrics dyed with the disperse dyes of the dyes 1, 4 and 5 increase with the increase of the dispersing agent concentration and reach their highest values at a concentration of 1.5 %. The K/S values for the polyester fabric dyed with the disperse dyes of the 2 and 3 dyes also increase with increasing dispersing agent concentration and reach their highest values at a concentration of 1%, while, the highest K/S value for dye No. 6 at a concentration of 0.5%. It is clear from the results described in table 1 that when dyeing polyester fabrics with dispersed dyes at a temperature of 100 °C or 130 °C the most appropriate optimum conditions for the use of dispersing agent are 1.5%.

Relation between* dyeing temperatures *and K/S

In our attempt to find the relationship between the color strength K/S and the temperature used in the dyeing process, table 2,3 and figure 2 revealed that the color strength K/S for high temperature dyeing at 130 degrees was higher than the color strength K/S for low temperature dyeing at 100 degrees, with rates ranging from 6 to 363%, for all dyes used except for dye No. 3, we got the opposite, but the difference between the two readings does not exceed 5%. From the above, we can say that high temperature dyeing is better than low temperature dyeing

Dyebath reuse of low and high dyeing temperatures.

We used the prepared disperse dyes in dyeing polyester fabrics at a temperature of 100 or 130 ^{0}C, we noticed that the dye residues contain a quantity of dye, so we used liquid dye wastes in dyeing undyed polyester fabrics for the purpose of treating these wastes and at the same time Obtaining dyed fabrics at almost no cost, which positively affects the environment. From the data obtained in table 4 that represented in figure 3, we observed that K/S value of the dyebath reuse process in the low temperature dyeing vary from 10-90% of its original value in the first dyeing process. Also, we observed that K/S value of the dyebath reuse process in the high temperature dyeing equal about 5-10% of its original value in the first dyeing process and this prove that high temperature dyeing is better than low temperature dyeing.

4. Fastness properties

The data listed in tables 5 and 6 showed that the fastness against washing, rubbing and perspiration gave very good results. The fastness properties against light gave acceptable results. Generally, the fastness properties of high temperature dyeing method are better than low temperature dyeing method.

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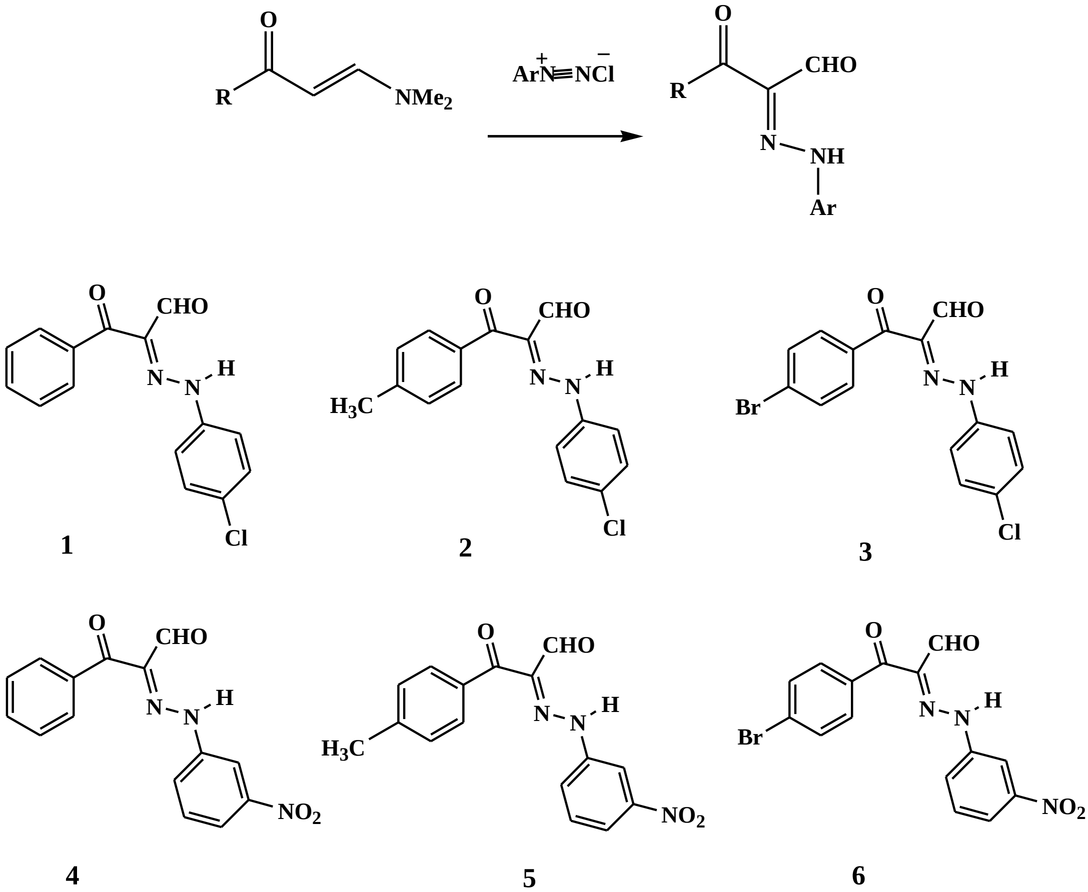

Figure 1. Chemical structures of the disperse dyes 1-6

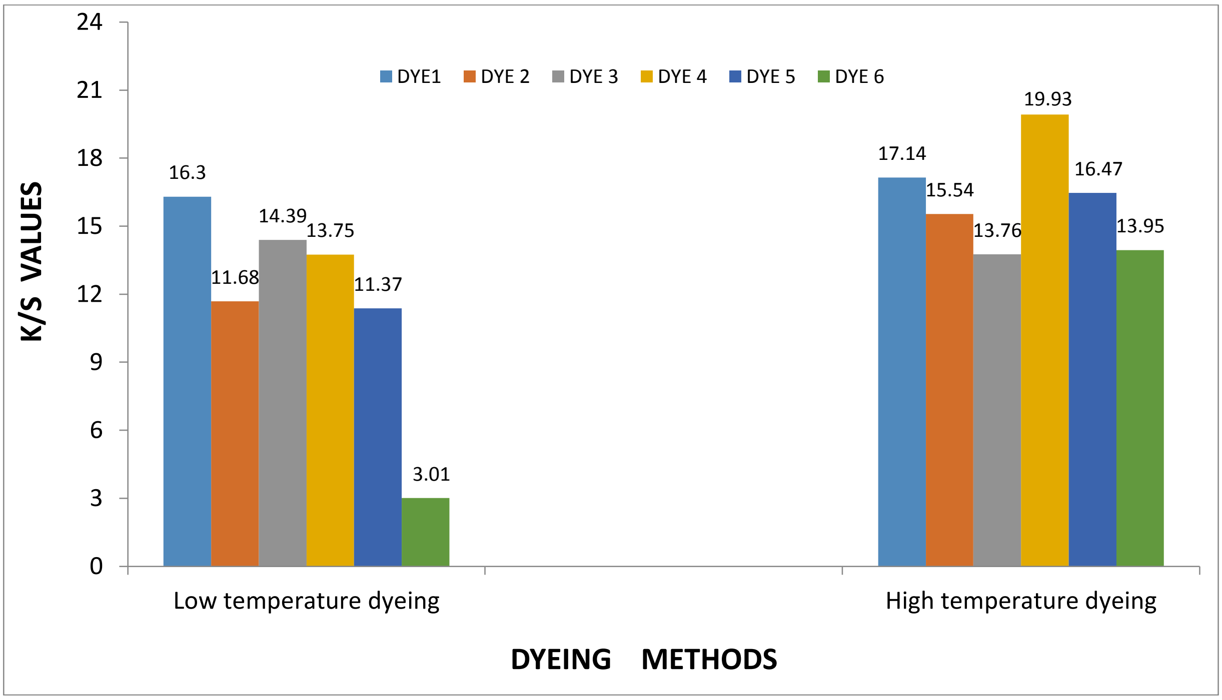

Figure 2.Relation between *K/S* and dyeing temperatures

Table 1: Dispersing agent effects on the dyeing temperature methods using3% shade.

Dye No	Dispersing agent %	K/S	
		Low temperature dyeing	Low temperature dyeing
	0.5%	9.46	11.80
	1%	9.78	9.94
1	1.5%	10.85	13.18
	2%	10.36	10.88
	0.5%	9.41	12.34
2	1%	9.89	16.99
	1.5%	8.48	11.63
	2%	10.23	15.85
	0.5%	9.29	9.43
3	1%	9.31	10.96
	1.5%	10.67	10.31
	2%	10.24	10.60
	0.5%	9.37	7.48
4	1%	7.88	9.53
	1.5%	7.15	10.27
	2%	10.36	9.47
	0.5%	8,64	11.13
5	1%	9.61	9.03
	1.5%	13.01	12.41
	2%	9.89	8.57
	0.5%	4.44	10.60
6	1%	6.62	4.67
	1.5%	7.54	9.22
	2%	7.29	7.48

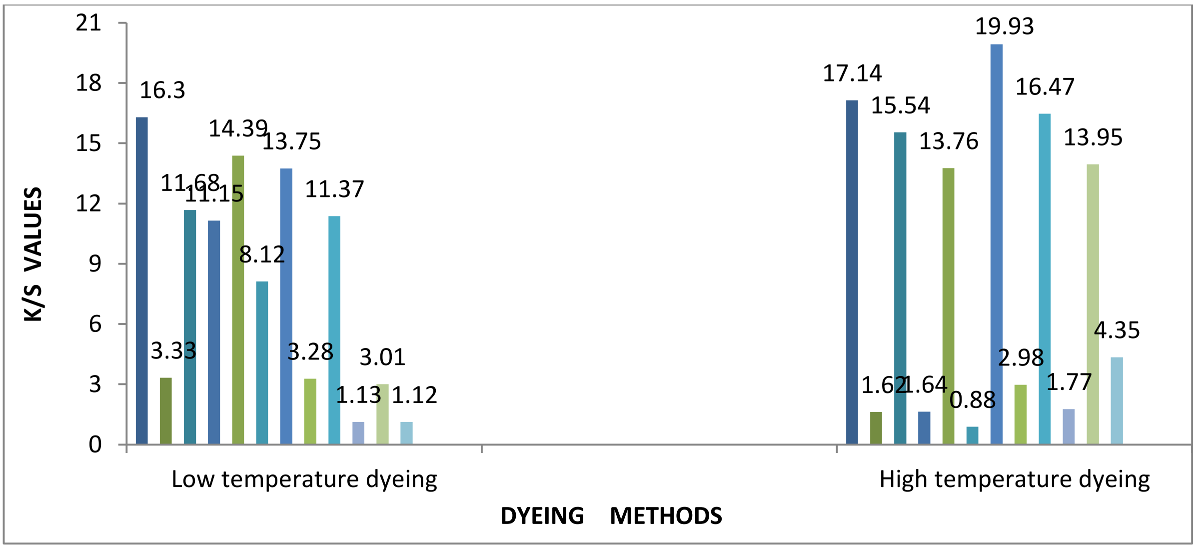

Figure 3.Relation between *K/S* and dyeing methods

Table 2: K/S of dyed fabrics at 100 ^{0}C.

Dye No	L*	*a**	*b**	*C**	*h**	K/S
1	83.94	-1.03	83.13	83.14	90.71	16.30
2	86.45	-8.60	67.57	68.11	97.26	11.68
3	87.12	-3.89	78.98	79.08	92.82	14.39
4	81.17	-0.23	51.38	51.38	90.26	13.75
5	83.62	-1.38	50.11	50.12	91.57	11.37
6	85.30	-2.29	37.78	37.85	93.47	3.01

Table 3: : K/S of dyed fabrics at 130 ^{0}C.

Dye No	L*	*a**	*b**	*C**	*h**	K/S
1	83.26	-0.12	83.09	83.09	90.08	17.14
2	85.47	-7.64	75.87	76.26	95.75	15.54
3	86.63	-9.02	75.02	75.56	96.86	13.76
4	78.44	5.33	59.76	60.00	84.91	19.93
5	81.06	2.85	57.38	57.45	87.15	16.47
6	79.00	-3.11	47.46	47.57	93.84	13.95

Table 4: Dyebath reuses of low and high dyeing temperatures

Dye No	K/S of Low temperature dyeing at 100 ^{0}C		K/S of High temperature dyeing at 130 ^{0}C	
	First dyeing	Dye reused	First dyeing	Dye reused
1	16.30	3.33	17.14	1.62
2	11.68	11.15	15.54	1.64
3	14.39	8.12	13.76	0.88
4	13.75	3.28	19.93	2.98
5	11.37	1.13	16.47	1.77
6	3.01	1.12	13.95	4.35

Table 5: Fastness properties of polyester fabrics dyed at 100 ^{0}C

Dye No	Washing fastness			Rubbing fastness		Perspiration fastness						Light fastness
						Acidic			Alkaline			
	SC	SW	Alt	Dry	Wet	SC	SW	Alt	SC	SW	Alt	
1	4−5	4	4−5	4−5	4	4−5	4	4	4−5	4	4	2−3
2	4−5	4	4−5	4−5	4	4−5	4−5	4	4−5	4	4	2−3
3	4−5	4	4−5	4−5	4	4−5	4	4	4−5	4	4	2−3
4	4−5	4	4−5	4	4	4	4	4	4−5	4−5	4	2−3
5	4−5	4	4−5	4−5	4	4−5	4−5	4	4−5	4	4	2−3
6	4−5	4	4−5	4−5	4	4−5	4	4	4−5	4−5	4	3−4

Table 6: Fastness properties of polyester fabrics dyed at 130 ^{0}C

Dye No	Washing fastness			Rubbing fastness		Perspiration fastness						Light fastness
						Acidic			Alkaline			
	SC	SW	Alt	Dry	Wet	SC	SW	Alt	SC	SW	Alt	
1	4−5	4	4	4−5	4	4−5	4	4	4−5	4	4	2−3
2	4−5	4	4	4−5	4	4−5	4−5	4	4−5	4−5	4	2−3
3	4−5	4	4	4−5	4	4−5	4−5	4	4−5	4	4	2−3
4	4−5	4	4	4−5	4	4−5	4−5	4	4−5	4	4	3−4
5	4−5	4	4	4−5	4	4−5	4	4	4−5	4	4	2−3
6	4−5	4	4	4−5	4	4−5	4	4	4−5	4−5	4	3

4. Conclusions

The disperse dyes were synthesized by us were utilized in dyeing polyester fabrics at low and high temperature, The most suitable conditions for the use of a dispersion agent are 1.5% when dyeing polyester fabrics with disperse dyes at a temperature of 100 ° C or 130 ° C. The color strength K/S and fastness properties of high temperature dyeing method are better than low temperature dyeing method where, the fastness against washing, rubbing and perspiration gave very good results while the fastness properties against light gave acceptable results.

Conflicts of interest

There are no conflicts to declare.

References

1- El-Apasery, M. A.; Yassin, F. A.; Abd El-Azim, M. H. M. ; Abdellatif, M. E. A. Synthesis and Characterization of some Disperse Dyes based on Enaminones, *J. Text. Color. Polym. Sci.*,**2020**, Vol. 17(1), 1-5.

2- Saleh, M. O.; El-Apasery, M. A.; Hussein, A. M.; El-Adasy, A.B.A.; Kamel. M. M. Microwave assisted synthesis of some azo disperse dyes part 2: Eco-friendly dyeing of polyester fabrics by using microwave irradiation , *European Journal of Chemistry*, **2021**, 12 (1), 64-68.

3- Iskender, M. A.; Becerir, B.; Koruyucu, A. Carrier Dyeing of Different Energy Level Disperse Dyes on Polyester Fabric, *Textile Res. J*. **2005**, 75(6), 462- 465.

4- Alnajjar, A., Abdelkhalik, M. M., Al-Enezi, A., Elnagdi, M. H. Enaminones as building blocks in heterocyclic syntheses: Reinvestigating the product structures of enaminones with malononitrile. A novel route to 6-substituted-3-oxo-2,3- dihydropyridazine-4-carboxylic acids. *Molecules*,**2009**, 14(1), 68-77.

5- Al-Mousawi, S. M, El-Apasery, M. A, Elnagdi, M. H. Green methodologies in organic synthesis: Microwave assisted solvent-and catalyst-free synthesis of enaminones and their conversion into 1,3,5-trisubstituted benzenes as well as 3-aroyl6-substituted pyridines. *European Journal of Chemistry*,**2011**, 2(2), 168-172.

6- Simunek, P.; Machacek, V. The structure and tautomerism of azo coupled b - Enaminones.*Dyes and Pigments*, **2010**,86(3), 197-205.

7- El-Apasery, M. A., Al-Mousawi, S. M., Mahmoud, H., Elnagdi, M. H. Novel Routes to Biologically Active Enaminones, Dienoic Acid Amides, Arylazonicotinates and Dihydropyridazines under Microwave Irradiation. *International Research Journal of Pure & Applied Chemistry*, **2011**,1(3), 69-83.

8- Al-Etaibi, A.M.; El-Apasery, M.A.; Ibrahim, M.R.; Al-Awadi, N.A. A facile synthesis of new monoazo disperse dyes derived from 4-hydroxyphenylazopyrazole-5-amines: evaluation of microwave assisted dyeing behavior. *Molecules*. **2012**, 17, 13891-13909.

9- Al-Etaibi, A. M.; Alnassar, H. S.; El-Apasery, M. A. Dyeing of polyester with disperse dyes: part 2. synthesis and dyeing characteristics of some azo disperse dyes for polyester fabrics. *Molecules*. **2016**, 21, 855-861.

10- Ashkar, S.M.; El-Apasery, M.A.; Touma, M.M.; Elnagdi, M.H. Synthesis of some novel biologically active disperse dyes derived from 4-methyl-2,6-dioxo-1-propyl-1,2,5,6-tetrahydropyridine-3-carbonitrile as coupling component and their colour assessment on polyester fabrics. *Molecules*. **2012**, *17*, 8822-8831.

11- Al-Etaibi, A. M. El-Apasery. M. A. Dyeing Performance of Disperse Dyes on Polyester Fabrics Using Eco-Friendly Carrier and Their Antioxidant and Anticancer Activities, *International journal of environmental research and public health*,**2019**, 16 (23), 4603.

12- Al-Saleh, B.; El-Apasery, M.A. Studies with condensed aminothiophenes: Further investigation on the reactivity of condensed aminothiophenes toward electron poor olefins and acetylenes under microwave heating. *J. Het. Chem*. **2006**, 43, 559–564.

13- Al-Saleh, B .; Behbehani, H.; El-Apasery, M. A.; Einagdi, M. H. Enaminones as building blocks in heterocyclic synthesis. Synthesis of polyfunctionally substituted 3-azolylpyridines and azolylazoloazines by thermal and microwave heating, *Journal of Chemical Research*,**2004**, (8), 575-577.

14- Al-Saleh, B.; Abdelkhalik, M.M.; El-Apasery, M.A.; Elnagdi, M.H. Studies with condensed thiophenes: Reactivity of condensed aminothiophenes toward carbon and nitrogen electrophiles. J. Chem. Res. **2005**, 23–26.

15- El-Apasery, M. A.; Hussein, A. M.; El-Adasy, A. A. M.; Saleh, M. O.; Kamel, M. M. Microwave assisted synthesis of some azo disperse dyes with antibacterial activities. Part 1. *Egypt. J. Chem.***2019**, 62. 1253- 1259.

16- El-Apasery, M. A., Al-Mousawi, S. M., Mahmoud, H., Elnagdi, M. H. Novel Routes to Biologically Active Enaminones, Dienoic Acid Amides, Arylazonicotinates and Dihydropyridazines under Microwave Irradiation. International Research Journal of Pure & Applied Chemistry, **2011**,1(3), 69-83.

17- Shawali, A. S. Synthesis, reactions and antitumour screening of new enaminones. Journal of Chemical Research, **2010**,34(11), 630-634.
18- Al-Qalaf, F., Almohammad, K., El-Apasery, M. A., Mahmoud, H. Synthesis of some biologically active monoazo disperse dyes derived from nicotinic acid derivatives under microwave irradiation for dyeing polyester fabrics. Eur. J. Chem. **2013**,4, 211-215.
19- Al-Omran, F., El-Khair, A. A. Heterocyclic synthesis via enaminones: novel synthesis of (1H)-pyridin2-one, pyrazolo[1,5-a]pyrimidine and isoxazole derivatives incorporating a N-methylphthalimide and their biological evaluation. Journal of Heterocyclic Chemistry, **2005**,42(2), 307-312.
20- El-Apasery, M.A.; Saleh, M.O. Monoazo disperse dyes based on pyrazolopyrimidinones provide added value. *Egypt. J. Chem.* **2021**, *64*, 4669–4674.
21- Al-Etaibi, A.M.; El-Apasery, M.A. Nano TiO_2 Imparting Multifunctional Performance on Dyed Polyester Fabrics with some Disperse Dyes Using High Temperature Dyeing as an Environmentally Benign Method. *Int. J. Environ. Res. Public Health* **2020**, *17*, 1377.