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Novel Synthesized Disperse Dyes based on Enaminones Provide Added-Value: Part 2

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Abstract
The synthesis of a number of enaminone-based disperse dyes is presented in this review article paper, and we describe in detail how these dyes have added value beyond simply being useful for dyeing polyester materials; In addition, these dyes have the ability to combat some types of gram-positive and gram-negative bacteria and some types of cancer.
***Keywords*:** Disperse dyes, Antibacterial activities, anticancer activity

1- Introduction

The demand for the use of enaminone derivatives has increased greatly because these compounds are involved in the synthesis of many different chemical compounds [1-36]. No one ever denies the role of these derivatives in the field of multifunctional organic chemistry, including textile chemistry [2-10]. Enaminones have been used as intermediates in the design of various naturally dynamic chemicals and the development of high-performance post-functionalization technologies for ingredients, including dental resins, ceramics, and coatings [44].

2. Chemistry

Here we clearly review that Scheme 1 illustrates the mechanism of reaction of acetophenone derivatives in the presence of xylene as a solvent with dimethylformamide dimethyl acetal to produce multiple derivatives with excellent yields of enaminones. We review how these enaminone derivatives are combined with diazonium chloride to produce azo dyes 1a-f with very excellent yields in an easy and promising way. Finally, we present how acetone is used to complete the cyclization process to react with compounds 1a-f to form new dispersion dyes 5a-f. The chemical composition of the new dyes was confirmed by elemental analysis, mass spectrometry, ^{1}H NMR spectra and (FT-IR) (see Scheme 1) [2].

3.Characterizations

We were able to produce six new dispersed azo dyes. We used the transfer dyeing process, which uses low temperatures. It is believed that transfer dyeing is one of the most important methods for dyeing polyester fabrics, as the transfer helps increase the absorption value of the dyes on the fabric. The carrier also plays an important role in dyeing delicate fabrics, which require less heat to dye because heat exceeding 100 degrees Celsius may damage these types of fabrics. The six pigments produce distinctive yellow to brown colors

4.Cytotoxicity activities

At the Al-Azhar University regional centre for mycology and biotechnology, cytotoxic effects against hepatocellular carcinoma and lung cellular carcinoma cells were demonstrated. The half-life (IC_{50}) of a substance required to cause 50% of healthy cells to become damaged. Assays for in *vitro* cytotoxicity should have several advantages, such as automation potential, reduced expenses, and speed. Furthermore, certain in vivo animal experiments may not be as relevant as testing done on human cells. However, there are still certain disadvantages associated with animal testing because they lack the technology innovation to fully replace it (table 1, figures 1-12) [5].

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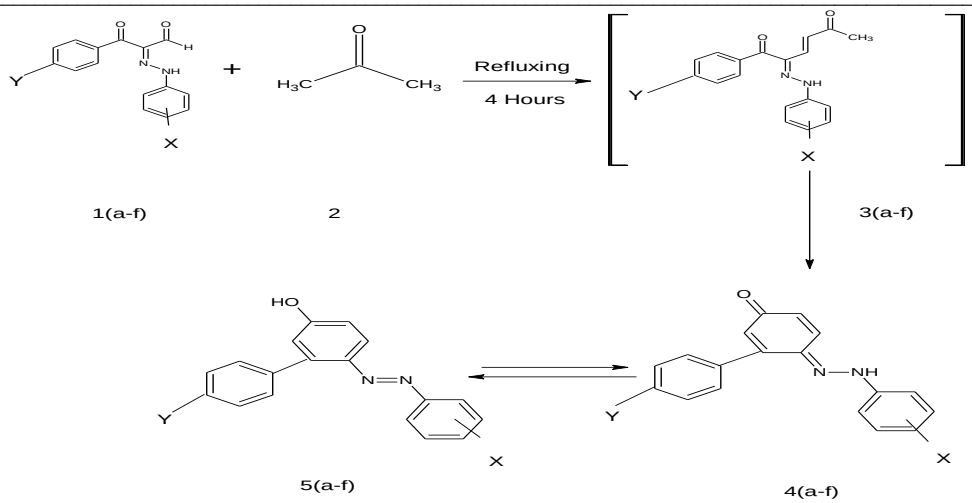


5 (a) X,Y= CL, H
5 (b) X,Y=CL,CH_3
5 (c) X,Y=CL, Br
5 (d) X,Y=No_2,H
5 (e) X,Y=No2,CH_3
5 (f) X,Y=No_2,Br

Scheme 1 : Chemical structure of disperse dyes [2]

Table (1) ***Cytotoxicity activities*** **of dyes**

Dye NO.	Cytotoxic activity (IC_{50}µg/ml) A-549	Cytotoxic activity (IC_{50} µg/ml) HepG-2
Dye 5a	41.93 µg/ml.	30.63 µg/ml.
Dye 5b	58.92 µg/ml.	47.93 µg/ml.
Dye 5c	51.69 µg/ml.	43.59 µg/ml.
Dye 5 d	41.91 µg/ml.	27.80 µg/ml.
Dye 5 e	186.65 µg/ml.	122.09 µg/ml.
Dye 5 f	28.22 µg/ml.	14.41 µg/ml.

The interaction of azo dyes with cells, or more precisely, the metabolites generated upon reduction of the azo link, is responsible for the cytotoxic effects of azo dyes. Metabolite damage can affect the structure and function of the DNA molecule. The list of medications' inhibitory effects against hepatocellular carcinoma (HEPG-2) cell lines that have been tested in vitro is descending. Dyes 5(f) > dye 5(d) > dye 5(a) > dye 5(c) > dye 5(b) > dye 5(e), according to the data in Table 1. The medicines' in vitro inhibitory activities against lung cancer cell lines (A-549) are listed in descending order as follows: Colour 5 (f) > Colour 5 (d) > Colour 5 (a) > Colour 5 (c) > Colour 5 (b) > Colour 5 (e)

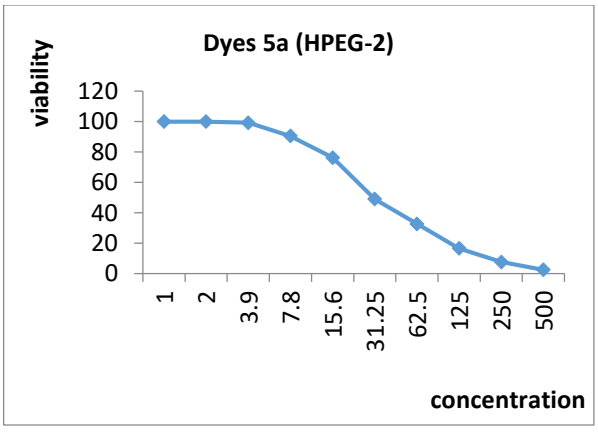


Figure (1)

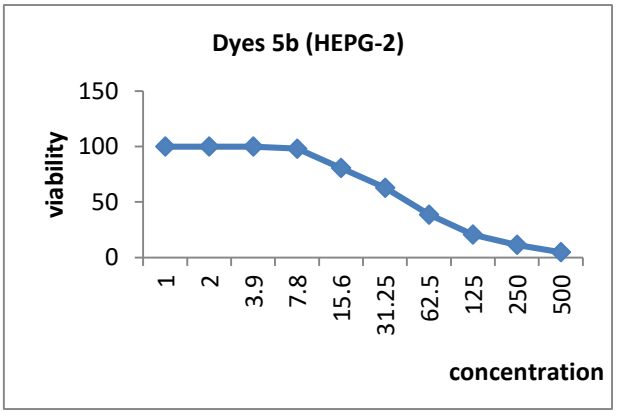
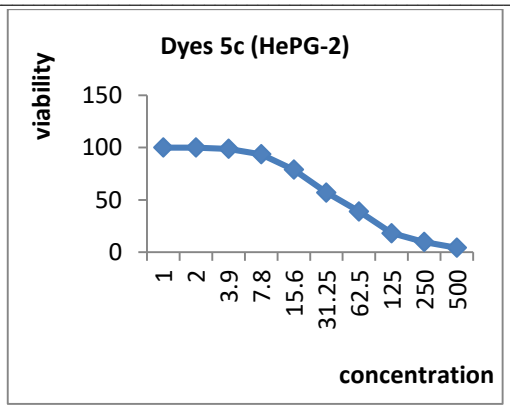


Figure (2)



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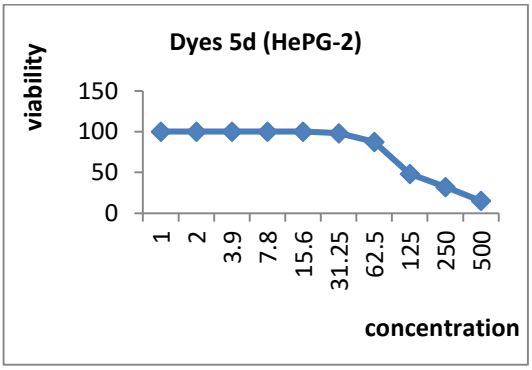

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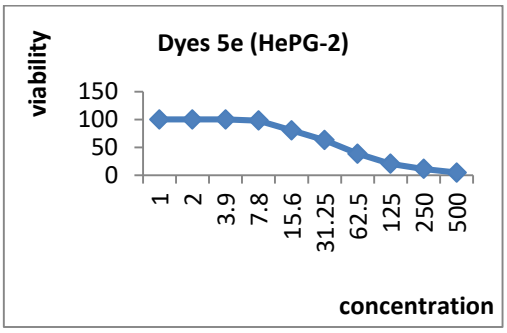

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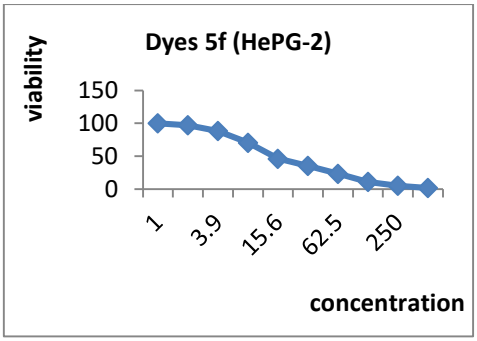

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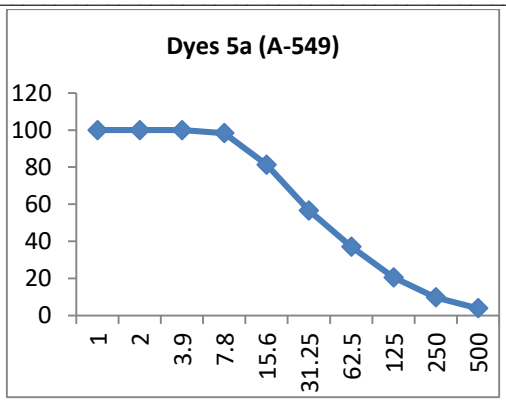

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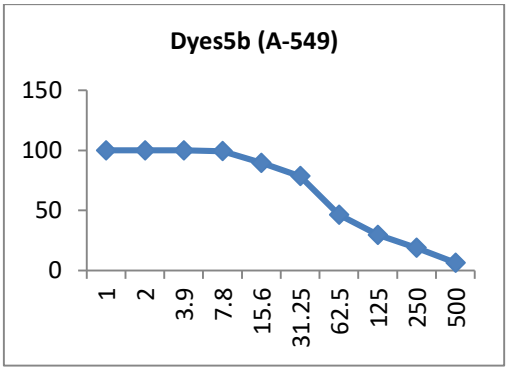

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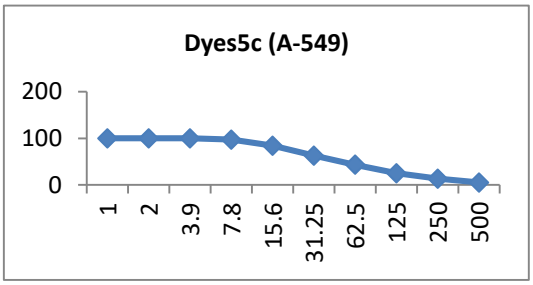

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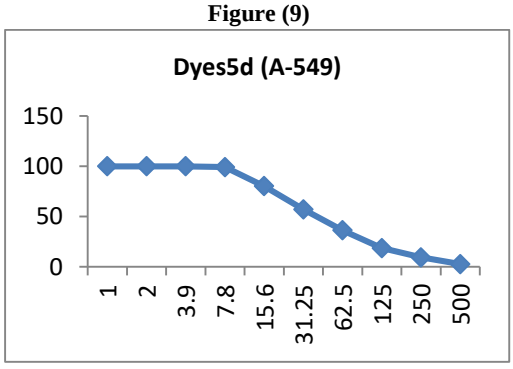

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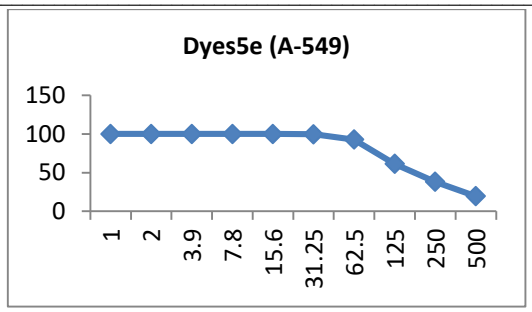

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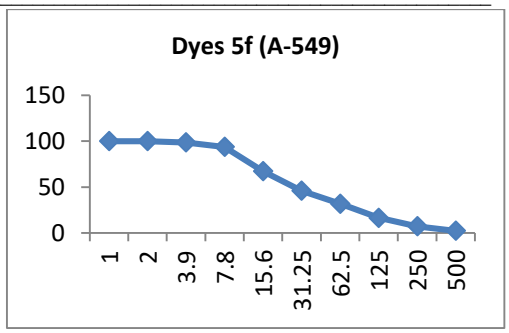

Figure (12

3.Antimicrobial activity of the dyes 5a-f

Compounds having groups that give or withhold electrons at the ortho, meta, and para regions of the hydrophobic aryl ring are more reactive than other derivatives, according to studies on the structure-activity relationship. Therefore, alterations in the acceptor side's hydrophobic

domain may be the cause of the compound's promising characteristics. The biological activity of six dyes was assessed using the diffusion agar method against four different microbiological cultures, with a measured volume of 100 l and a well diameter of 6.0 mm. Aspergillus fumigatus, Candida albicans, and pure cultures of Escherichia coli, a Gram-positive and Gram-negative bacterium, and Staphylococcus auerus, a Gram-positive bacterium, were used. Ketoconazole was used as a positive control for the fungus (gentamycin).(table 2) [2].

Table 2:-Antimicrobial activity of the dyes 9a-f.

Dye Number / Microorganisms	Inhibition zone diameter (Nearest mm)						Ref.
	Dye 5a	**Dye 5b**	**Dye 5c**	**Dye 5d**	**Dye 5e**	**Dye 5f**	
Aspirgillus fumigatus	16	15	19	NA	17	NA	2
Candida alpicans RCMP 005003(1)ATCC 10231	28	13	18	16	15	26	
Staphylococcus aureus (RCMP010010)	24	12	16	14	14	23	
Escherichia coli (RCMP 010052) ATCC 25955	15	12	12	NA	NA	NA	

The materials were tested at a concentration of 10 mg/mL, and the mean zone of inhibition was measured in millimetres above the well diameter at the National Research Centre. The results are shown in Table 2. The inhibitory zones shown in that table show that the dyes **5a–f** efficiently inhibits at least two of the pathogens being studied. In addition to showing strong activities against *Staphylococcus aureus* at 24 mm and *Escherichia coli* at 15 mm.

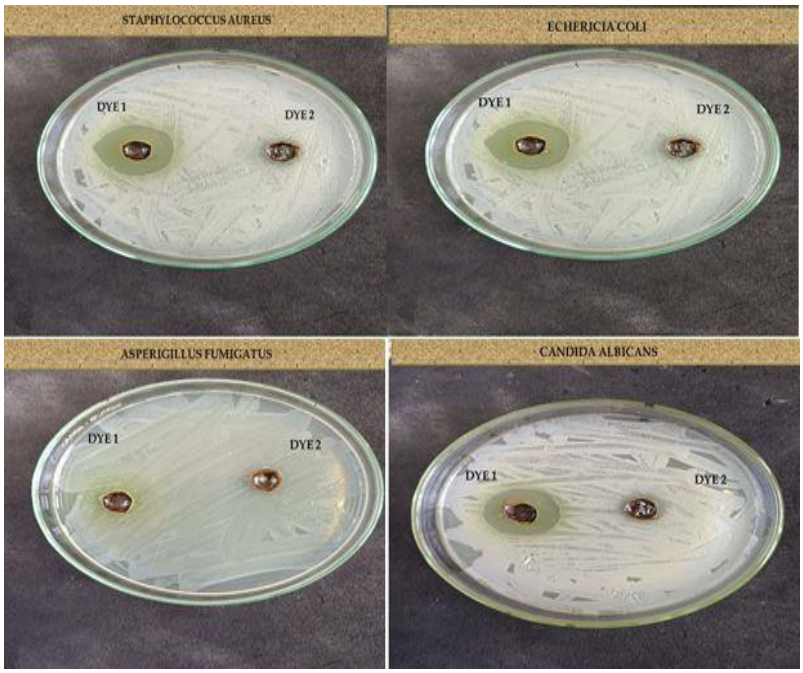

Figure 13: Biological activities of dyes 5a,b

Dye **5a** also shows strong activities against *Aspirgillus flavus* and *Candida alpicans* fungi (c.f. Figure 13).
Dye **5b** exhibits strong activities with a significant inhibition zone of 15 mm, moderate activities of 13 mm against *Staphylococcus aureus*, and strong activities of 12 mm against *Escherichia coli* (c.f. Figure 13).
Dye **5c** shows strong activities with a significant inhibition zone of 19 mm against *Aspirgillus flavus*, moderate activities of 18 mm against *Candida alpicans* , moderate activities of 16 mm against *Staphylococcus aureus*, and strong activities of 12 mm against, *Escherichia coli* (c.f. Figure 14).

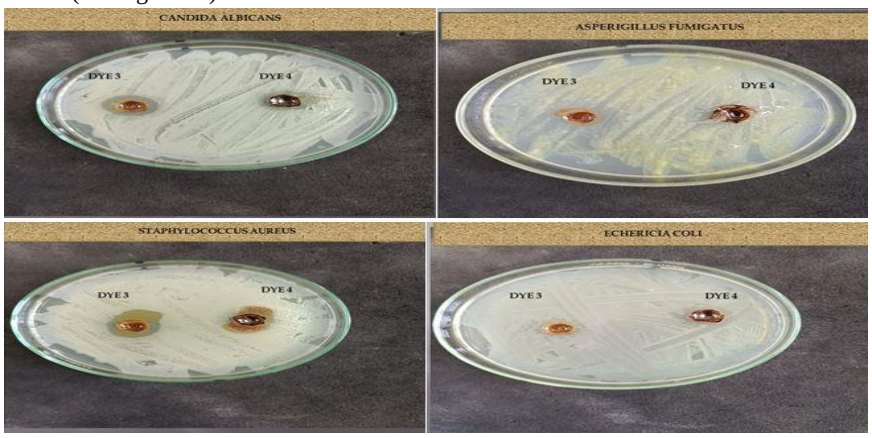

Figure 14: Biological activities of dyes 5c,d

Figure 14 shows that Dye **5d** has no activity against *Aspirgillus flavus*, moderate activity equivalent to 16 mm against *Candida alpicans* fungi, moderate activity equal to 14 mm against *Staphylococcus aureus*, and no activity against *Escherichia coli*, with a notable inhibitory zone of 15 mm, Dye **5e** shows robust activity in relation to *Aspirgillus flavus*. On the other hand, it has moderate activity against *Staphylococcus aureus*, with a zone of 12 mm, and moderate activity against *Candida alpicans*, with a zone of 13 mm. Regarding *Escherichia coli* bacteria, no effect is seen. Only *Aspirgillus flavus*, is resistant to dye.**5f** doesn't do anything. Along with no action against *Escherichia coli*, strong activities *Staphylococcus aureus* and *Candida alpicans* measuring 23 mm are also shown (c.f. Figure 15).

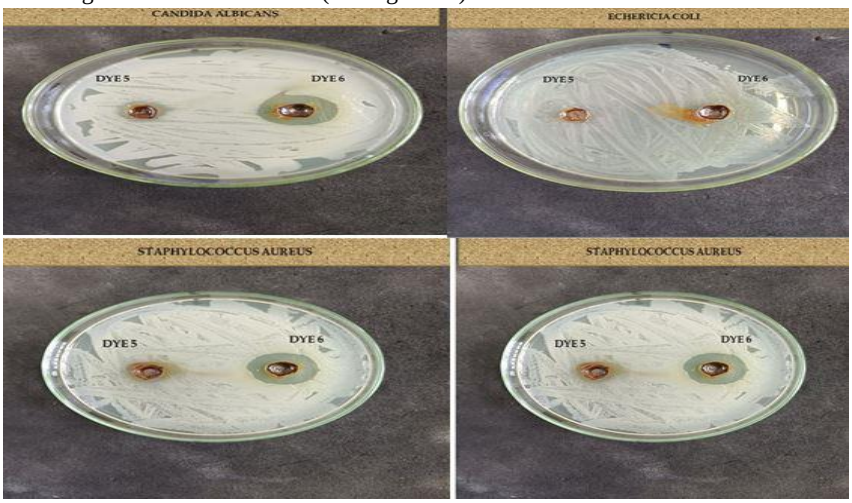

Figure 15: Biological activities of dyes 5 e,f

3.Conclusions

Our approach to the synthesis and characterisation of innovative disperse dyes is complemented by the creation of new disperse dyes and the confirmation of their chemical composition. We then dyed polyester clothes with these inventive dyes. It was also discussed how the antibacterial activity of the new dispersed dyes against different types of germs and malignancies led to positive outcomes for the use of these innovative dyes in pharmaceutical and medical context

References

[1] MA El-Apasery, ME Abdellatif, AM Ahmed, Novel Synthesized Disperse Dyes based on Enaminones Provide Added-Value: Part 1, Egyptian Journal of Chemistry **2024**, 67 (2), 185-197
[2] El-Apasery, M. A.; Abdellatif, M. E. A.; Yassin, F. A.; Ahmed, S. M. Synthesis of novel disperse dyes based on arylazophenols: Synthesis, Characterizations and applications. *Bull. Chem. Soc. Ethiop.* **2023**, 37(4), 993-1002.
[3] K Abdelmoteleb, MA El-Apasery, A Wasfy, Synthesis of New Disperse Dyes Based on Enaminones Derivatives: Part 2. Antioxidant Activities, Egyptian Journal of Chemistry, **2024**, 67 (6), 61-65
[4] Elapasery, M., Yassin, F., Abdellatif, M. Enaminones-Assisted Synthesis of Disperse Dyes. Part 3: Dyebath Reuse and Biological Activities', *Egyptian Journal of Chemistry*, **2020**, 63(9), 3503-23158.

[5] El-Apasery, M. A.; Abdellatif, M. E. A.; Yassin, F. A.; Ahmed, S. M. Synthesis of novel disperse dyes based on arylazophenols: Part 2. Anticancer Activities. *Egyptian Journal of Chemistry*, 2023, 66 (4), 49-53.
[6] MA El-Apasery et al. Effective Removal of Reactive Violet 2 Dye by Using Different Geopolymers Containing Bentonite, Egyptian Journal of Chemistry, **2024**, 67 (3), 467-474
[7] Elapasery, M..A, Yassin, F. A.; , Abdellatif, M. E. A. Nano ZnO Provides Multifunctional on Dyed Polyester Fabrics with Enaminone-Based Disperse Dyes. . *Egypt. J. Chem.* **2022**, *65*(6), 37–45.
[8] AM Al-Etaibi, MA El-Apasery, A Holistic Review of 3-Dimethylamino-1-Arylpropenones Based Disperse Dyes for Dyeing Polyester Fabrics: Synthesis, Characterization, and Antimicrobial Activities, Polymers, **2024**, 16 (4), 453
[9] 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.
[10] Elapasery, M., Yassin, F., Abd El-Azim, M., Abdellatif, M. Enaminones-Assisted Synthesis of Disperse Dyes. Part 1: Low Temperature Dyeing of Polyester Fabrics', *Egyptian Journal of Chemistry*, **2020**, 63(3), 1101-1108.
[11] Elapasery, M..A, Yassin, F. A.; , Abdellatif, M. E. A. Nano TiO_2 Provides Multifunctional on Dyed Polyester Fabrics with Enaminone-Based Disperse Dyes. *Egypt. J. Chem.* **2022**, *65*(6), 47–54.
[12] 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.
[13] El-Apasery, A.M.; Shakra, S.; Abbas, D.; Gaffer, H.; Allam, A. Synthesis of some azo disperse dyes based on pyridone moiety and their application on polyester fabrics. *Egypt. J. Chem.***2018**, *60*, 97–102.
[14] Shawali, A. S. Synthesis, reactions and antitumour screening of new enaminones. Journal of Chemical Research, **2010**, 34 (11) , 630-634.
[15] 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.
[16] 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(5). 1253-1259.
[17] La Maza, A. D.; Manich, A. M.; Coderch, L.; Parra, J. L. Multilamellar Liposomes Including Cholesterol as Carriers of Azobenzene Disperse Dyes in Wool Dyeing, Textile Res. J. 1995, 65(3), 163- 170.
[18] K Abdelmoteleb, AAF Wasfy, MA El-Apasery, Novel Disperse Dyes Based on Enaminones: Synthesis, Dyeing Performance on Polyester Fabrics, and Potential Biological Activities, Molecules, **2024**, 29 (10), 2227
[19] Simunek, P.; Machacek,V.The structure and tautomerism of azo coupled-Enaminones.Dyes and Pigments, **2010**, 86(3), 197-205.
[20] Kascheres, C. M. The chemistry of enaminones and small rings: our contribution. J.Braz.Chem.Soc.,**2003**,14(6),945-969.
[21] Abdelkhalik, M.M.; Negm, A.M.; Elkhouly, A.I.; Elnagdi, M.H. Studies with condensed aminothiophenes: Further investigation of reactivity of aminothienocoumarines and aminothienobenzo[h]coumarines toward electron-poor olefins and acetylenes. Heteroat. Chem. 2004, 15, 502–507.
[22] Gorobets, N.Y., Sedash, Y.V., Shishkina, S.V., Shishkin, O.V., Yermolayev, S.A., Desenko, S. M. Structure of the intermediate in the synthesis of 6-methyl-2-oxo-1,2-dihydropyridine-3-carbonitrile. Arkivoc, **2009**, 13, 23-30.
[23] Al-Awadi, N. A., Ibrahim, MR., Abdelhamid, I. A., Elnagdi, M. H. Arylhydrazonals as the aldehyde component in Baylis-Hillman reactions. Tetrahedron, 64(35), **2008**, 8202-8205
[24] El-Apasery, M. A. ; Al-Mousawi, S. M. ; Mahmoud, H.; and Elnagdi,M.H.Studies with Biologically Active Enaminones: An Easy Method for Structural Elucidation of Products Produced from Enaminone Starting Materials through Pathways Employing Microwave Irradiation, International Research Journal of Pure &Applied Chemistry, **2012**, 2(1), 77-90.
[25] Al-Mousawi, S. M., El-Apasery, M. A., Al-Kanderi, N. H. Microwave-assisted organic synthesis: the Gabriel approach as a route to new pyrazolylhydrazonoazoles. ARKIVOC, **2008**, 16, 268-278.
[26] 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.
[27] 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, 2005, 23–26.
[28] MA El-Apasery, ME Abdellatif, SM Ahmed, Synthesis of Novel Disperse Dyes based on Arylazophenols: Part 5. A Comparison between Dyeing Methods at Low and High Temperatures for Polyester Fabrics
[29] Egyptian Journal of Chemistry, **2024**, 67 (1), 371-374
[30] 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-aroyl-6-substituted pyridines. European Journal of Chemistry, **2011**, 2(2), 168-172.

[31] Al-Mousawi,S.M.,El-Apasery,M.A.,Elnagdi, M. H. Enaminones in heterocyclic synthesis: a novel route to tetrahydro pyrimidines,dihydropyridines, triacylbenzenes and naphthofurans under microwave irradiation.Molecules.**2010**,15,58-67.

[32] Al-Omran, F.; El-Khair, A. A. Heterocyclic synthesis via enaminones: novel synthesis of (1H)-pyridin-2-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.

[33] Al-Saleh, B., El-Apasery, M. A., Abdel-Aziz, R. S., Elnagdi, M. H.Enaminones in heterocyclic synthesis: Synthesis and chemical reactivity of 3-anilino-1-substituted-2-propene-1- one.Journal of Heterocyclic Chemistry, **2005**, 42(4), 563-566.

[34] El-Adasy,A.A.A.Kamel,M.M.;Saleh,M.O.;Hussein,A.M.;El-Apasery,M.A.Disperse Dyes Based on Pyrazolopyrimidinon es I: Their Dyeing Applications and Antimicrobial Activities. *Int. J. Chem. Tech. Res.* **2016**, *9*, 31-38.

[35] 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

[36] Al-Etaibi, A. M., Al-Awadi, N. A., El-Apasery, M. A., Ibrahim, M. R. Synthesis of some novel pyrazolo[1,5-a]pyrimidine derivatives and their application as disperse dyes. Molecules, **2011**, 16, 5182-5193.