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Synthesis, characterization and fluorescence properties of newly synthesized flavonoid derivatives from chalcone

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Abstract

In this work, substituted coumarin based flavonoids have been synthesized from resorcinol. The main purpose of that synthesis was to study fluorescence activity of synthesized compounds.The entire synthesized compounds were characterized by ^{1}H-NMR, IR, mass spectroscopic techniques. Melting points (M.P) and yields (%) of all the compounds are recorded

The reactions are easy to conduct, under mild conditions, and form coumarine substituted flavones in moderate to excellent yields.

Keywords: Chalcone, flavonoid, fluorescence activity.

Introduction

Flavonoids are the secondary metabolites of phenolic nature and are responsible for the variety of pharmacological activities in plants.[1-5]Flavonoids are phenolic substances and are known to be synthesized by plants in response to microbial infection.[6] Flavonoids are located in the nucleus of mesophyll cells and within centers of ROS generation. They also regulate growth factors in plants such as auxin.[7-10] The chemical effects of flavonoids depend on their structural class, conjugations, degree of polymerization & degree of hydroxylation and substitutions.[11] Recent research interest in these substances has been encouraged due to their potential health benefits arising from their antioxidant activities. Functional hydroxyl groups in flavonoids mediate their antioxidant effects by scavenging free radicals or by chelating metal ions.[12-16] In addition, flavonoids are thought to have health-promoting properties due to their high antioxidant capacity and thus act as a dietary component to induce human protective enzyme systems. The number of studies has suggested protective effects of flavonoids against many diseases caused by bacteria and virus. It also acts against degenerative diseases such as cardiovascular diseases, cancers, and other age-related diseases.[17-19]Flavonoids acts as flavorants, colorants and antioxidants and thus influence the quality and stability of foods.[20-21]antibacterial activity.[22-25] antimicrobial activity.[26]The corresponding substituted flavonoids are molecules of current interest as they have potent biological activity. [27-28]

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2. Material and method

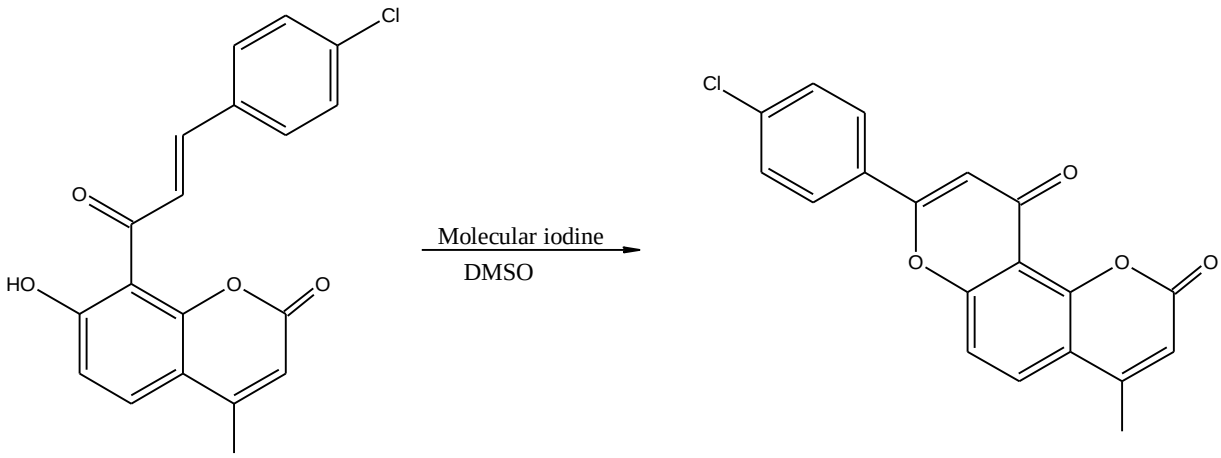
Melting points were determined on a Veego Melting Point Apparatus Mod. VMP-DS ± 0.5˚C accuracy and are uncorrected. The ^{1}H NMR spectra were recorded on a BRUKER Spectrometer (400 MHz) in DMSO solvent. Chemical shifts were reported in parts per million using tetramethylsilane as an internal standard and were given in δ units. Infrared spectra were taken on SHIMADZU-FTIR-8400 Spectrophotometer instrument in the frequency range of 4000-400 cm^{-1} by KBr powder method. The Mass spectra were recorded by MS-SHIMADZU-QP2010. All reactions were monitored by thin layer chromatography, carried out on 0.2 mm silica gel 60F254 (Merk) plates using UV light for detection. Common reagent grade chemicals are either commercially available and were used without further purification.

Representative method for the preparation of final compounds:

Few iodine crystals were added in the mixture of chalcone and DMSO. The reaction mixture was refluxed for 1 hr. to get the final products flavonoids. The solid product obtained was dissolve in ethanol and crystallized in hot ethyl alcohol. The progress and completion of reaction was monitored by thin layer chromatography (TLC) to obtain the final compounds.

: Preparation of 8-(4-chlorophenyl)-4-methyl-2H,10H-pyrano[2,3-f]chromene-2,10-dione (2a):

Few iodine crystals were added in the mixture of compound (**1a**) and DMSO. The reaction mixture was refluxed for 1 hr. to get the final product 8-(4-chlorophenyl)-4-methyl-2H,10H-pyrano[2,3-f]chromene-2,10-dione (2a).



1a 2a

It is brownish crystalline solid; yield 76%; mp.176-178^{0}C; IR spectrum (KBr), υ (cm^{-1}): 3235. cm^{-1}(Ar-CH): 2971. (Al-CH Str): 1567(Ar C=C str.); cm^{-1} 1682 cm^{-1} (C=O ketone str.); 1241 cm^{-1}(C-O-C str.);

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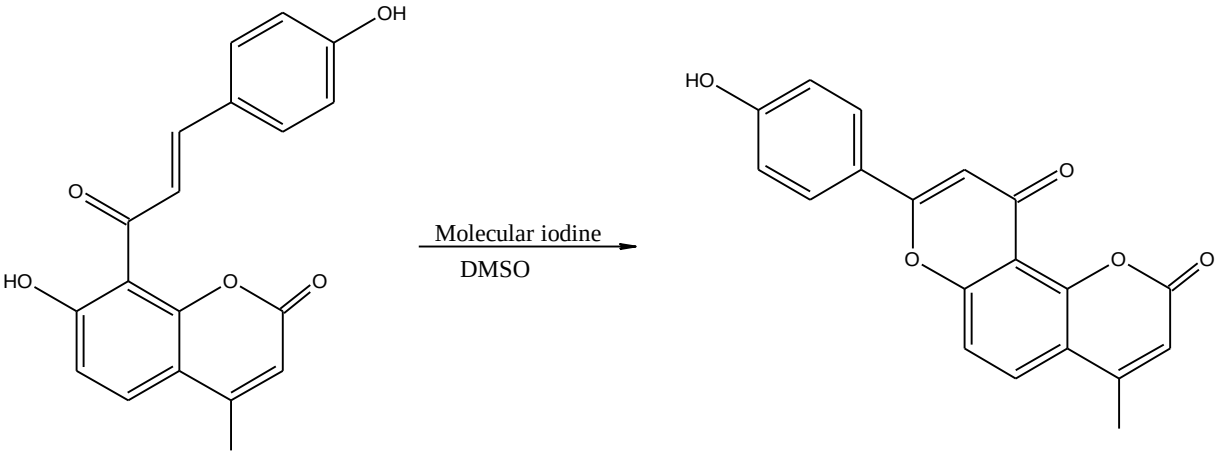
748.90 cm^{-1} (C-Cl str.); ^{1}HNMR spectrum (δ ppm): 2.3 (3H, s, CH_3); 6.01 (1H, d, CH); 5.96 (1H, s, CH); , 6.71 (2H, d, CH); 6.8 (2H, d, CH-); 7.2 (1H, d, Ar-H)-7.3 (d, 1H, Ar-H), 7.8 (1H, d, CH).

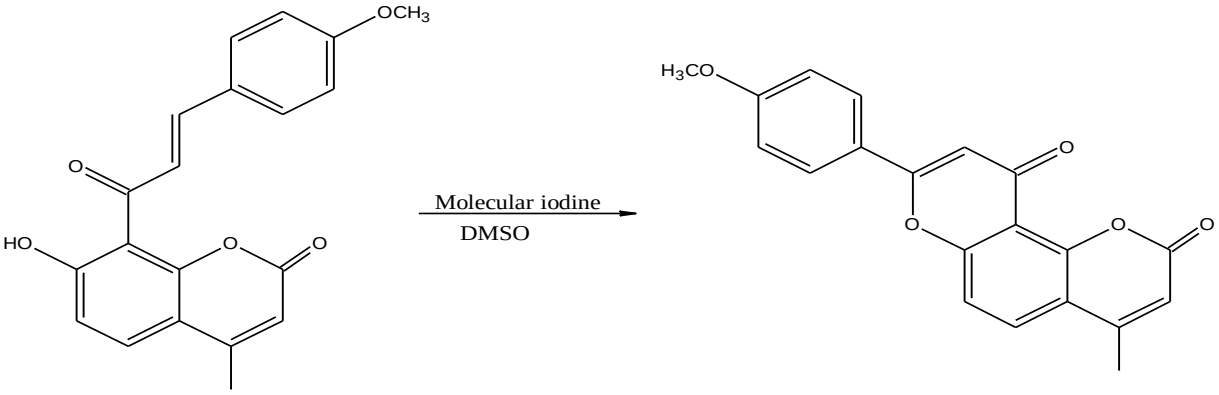
(4-hydroxyphenyl)-4-methyl-2H,10H-pyrano [2,3-f]chromene-2,10-dione (2b):



1b 2b

It is brownish crystalline solid; yield 74%; mp.176^{0}C; IR spectrum (KBr), υ (cm^{-1}): 1473.50 cm^{-1} (Ar C=C str.); cm^{-1} 1700.39 cm^{-1} (C=O ketone str.); 1263.90 cm^{-1}(C-O-C str.); 3468.90 cm^{-1} (O-H str.); ^{1}HNMR spectrum (δ ppm): 1. 72 (3H, s, CH_3); 6.01 (1H, d, CH); 5.90 (1H, s, CH); , 6.71 (1H, s, CH); 6.8 (1H, d, CH); 7.22-7.28 (m, 4H, Ar-H), 9.63 (1H, s, OH).

8-(4-methoxyphenyl)-4-methyl-2H,10H-pyrano [2,3-f]chromene-2,10-dione (2c):



1c 2c

It is brownish crystalline solid; yield 74%; mp.178-180^{0}C; IR spectrum (KBr), υ (cm^{-1}): 1473.52 cm^{-1} (Ar C=C str.); cm^{-1} 1700.39 cm^{-1} (C=O ketone str.); 1263.93 cm^{-1}(C-O-C str.); ^{1}HNMR spectrum (δ

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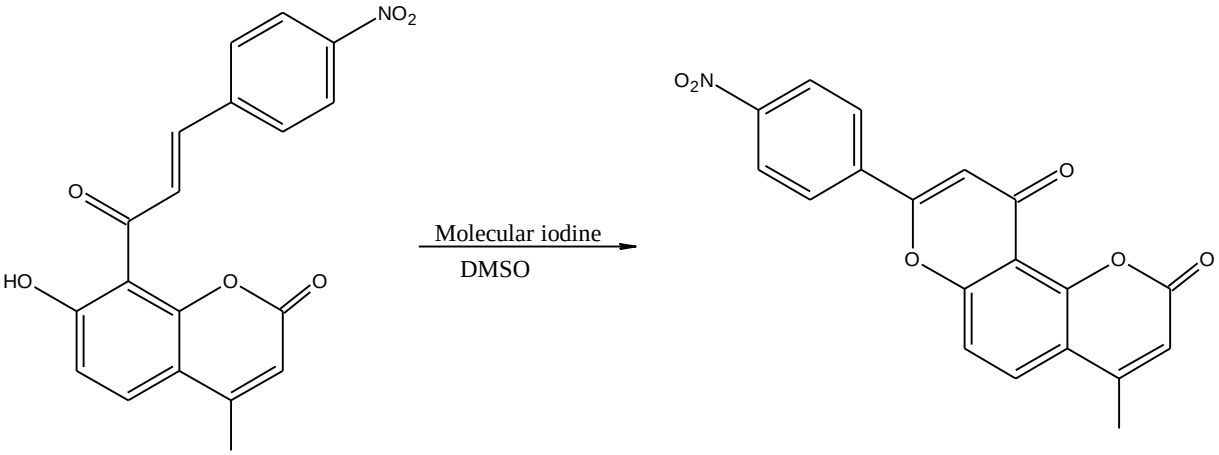
ppm): 2. 23 (3H, s, CH_3); 2.55 (3H, s, CH_3); 6.01 (1H, s, CH); 6.8 (1H, s, CH);6.71 (1H, d, CH);6.7 (1H, d, CH); 7.4-7.5 (4Ar-H).

8-(4-nitrophenyl)-4-methyl-2H,10H-pyrano[2,3-f]chromene-2,10-dione (8d):



1d 2d

It is brownish crystalline solid; yield 68%; mp. 167 ^{0}C; IR spectrum (KBr), υ (cm^{-1}): 1474.23 cm^{-1} (Ar C=C str.); 1700.97 cm^{-1} (C=O ketone str.); 1267.08 cm^{-1}(C-O-C str.); 1344.21/1600.43 cm^{-1} (NO_2 str.); ^{1}HNMR spectrum (δ ppm): 2. 67 (3H, s, CH_3); 7.02 (1H, d, CH); 7.28 (1H, d, CH); 6.28 (1H, s, CH); 6.73 (1H, s, CH);7.58-7.72 (4H, Ar-H).

Result and discussion:

3.1 Fluroscence properties:

The fluorescence properties of uniqueflavonoids 2**a-d** were investigated. The effect of different substituent's on flavonoid moiety and different solventswith respect to polarity has been studied. The molecules were designed with unique combination of electron donor at the 7th position like benzene substituted with chloro, hydroxy, nitro, methoxyetc coumarin moiety leading to the formation of push pull system.

The fluorescence spectral data of all the compounds 2a-d are summarized in Table 1. These compounds exhibited varying trend of fluorescent property with29 to 64 nanometer strokes shift in chloroform, Acetonitrile, methanol and DMSO respectively when compared with Rhodamine B.

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Table 1. Fluorescent property of compound 2a-d in four solvent viz. MeOH, Acetonitrile,Chloroform dimethylsulphoxide.

Compounds	Solvent	λabs (nm)	λem (nm)	Strokes shift
2a	Chloroform	402	437	35
	Acetonitrile	427	467	40
	Methanol	379	427	48
	DMSO	434	476	42
2b	Chloroform	388	422	34
	Acetonitrile	424	469	45
	Methanol	340	404	64
	DMSO	448	487	39
2c	Chloroform	401	435	34
	Acetonitrile	410	451	41
	Methanol	411	455	44
	DMSO	440	469	29
2d	Chloroform	444	475	31
	Acetonitrile	425	466	41
	Methanol	416	455	39
	DMSO	438	478	40

The absorption, emission and strokes shift are also given in Table 1.

Antibacterial activity

Antibacterial activity of synthesized flavonoids has been examined. The antibacterial activity against five bacterial species has been studied. The compounds show moderate activity against bacterial species and are summarized in table 2.

Sr. no.	Antibacterial activity				
	Diameter of zone of inhibition (in mm)				
	Gram posetive		*Gram negative*		
	S. aureus	*S.pneumoniae*	*S.typhi*	*E. coli*	*Pseudomonas*
2a	4	4	6	3	9
2b	4	3	7	3	9
2c	6	3	6	3	10
2d	7	5	11	5	16

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Conclusion

The successful synthesis of flavonoid compounds follows a mild, efficient route with a good to moderate yield. In present work we synthesized flavonoids from early prepared chalcone. The synthesized compounds exhibited competent and satisfactory fluorescence activity as well as antibacterial activity.

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