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Review Article

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Comparative Study of Microwave and Conventional Synthesis and Pharmacological activity of Coumarins : A Review

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ABSTRACT

Coumarins have been well known naturally occurring oxygen heterocyclic compound isolated from various plants. The plant extracts containing coumarin–related heterocycles are employed as herbal remedies in traditional synthesis of medicine. Coumarin is a phyto chemical (benzopyrone) a toxin found in many plants, notably in high concentration in the tonka bean, vanilla grass, woodruff , mullein, levender, licorice, strawberries, apricots, cherries, cinnamon, sweet clover and bison grass having vanilla like flaver. Due to the potential application in fragrance, pharmaceutical and agrochemical industries the coumarins occupy an important position in natural and synthetic organic chemistry. Synthesis of coumarins and their derivatives has attracted considerable attention from organic and medicinal chemists for many years as a large number of natural products contains this heterocyclic nucleus. Coumarins comprise a vast array of biologically active compounds with several types of pharmaceutical agents possessing anticancer, anti-HIV, anticoagulant, spasmolytic and antibacterial activity, cytotoxic activity in vitro and in vivo. This review is based on recent studies of coumarin and coumarin related compound and comparison of conventional and microwave irradiation showed the later procedure required shorter reaction times, generally gave higher yield and was applicable to a larger set of substrate.

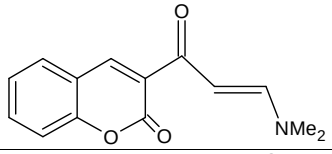
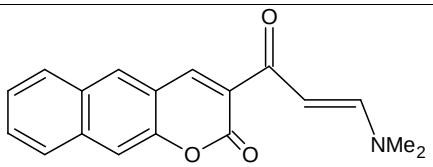
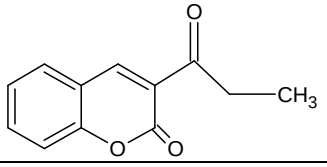
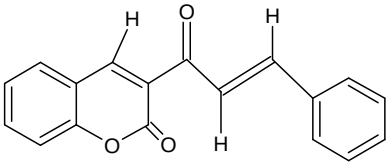
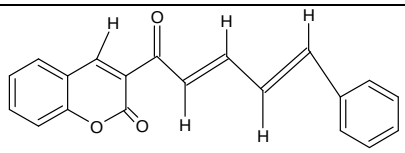
Key words: Coumarin, microwave synthesis, pharmacological activities, SAR, various synthesis.

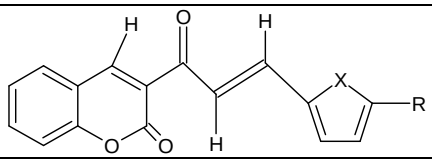
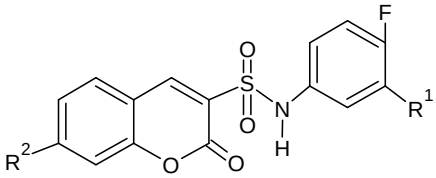
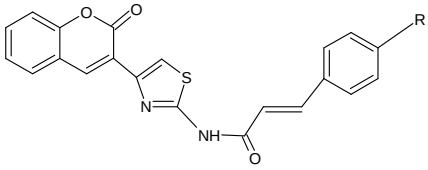
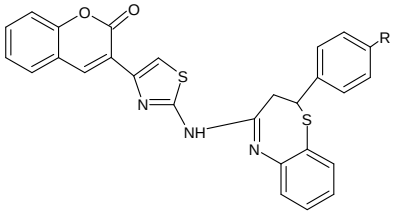
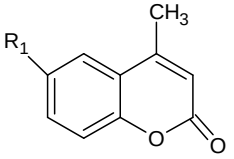
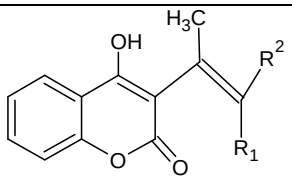
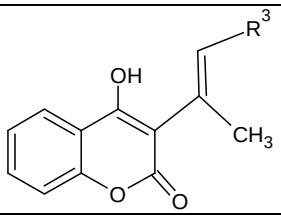
INTRODUCTION

Coumarins (2H-1-bengopyran-2-ones) are important oxygen containing fused heterocycles used in drugs and dyes[1]. Coumarins be bound their class name to ‘coumarou’ the vernacular name of the Tonka bean (*Dipteryx odorata willd, Fabaceae)*, from which coumarin itself was isolated in 1820[2]. They are the family of lactones containing benzopyrone skeletal framwork that have enjoyed isolation from plant as well as total synthesis in the laboratory[3]. The incorporation group as a fused component into parent coumarin alters the property of parent coumarin and converts it into a more useful product[4]. Coumarin are plant flavonoids widely distributed in nature. Natural coumarins are known to have antidiabetic activity[5], anabolic antioxidant and hepato protective activities[6]. Substituted coumarins derivatives have been reported to have variety of biological activities. The potent antibiotics like Novobiocin, Coumaromycin and Chartesium are coumarin derivatives. Recently, the interest on these compounds has been revived owing to their use as fluorescent markers in the biochemical determination of enzymes. Coumarin derivatives can be synthesized by one of such methods as the Claisen rearrangement[7], Perkin reaction[8], Pechmann reaction[9], Witting reaction[10], as well as the Knoevnagel condensation[11]. The plant

extract containing coumarin related heterocycles which were employed as herbal remedies in early days, have now been extensively studied for their biological activities[3]. Derivatives of coumarins usually occurs naturally as secondary metabolite present in seed, roots and leaves of many plant species[12]. Microwave irradiation has since been proven to be extremely useful for promoting and simplifying many condensation reactions which can be carried out both in solvent and under solvent free condition. The essence of this work was synthesis of coumarin derivatives using microwave irradiation in comparison with conventional methods. These investigation have revealed their potentials as versatile biodynamic agent for example-3-heteroaryl substituted coumarin and benzocoumarins of potential interest as pharmaceuticals and photochromic dyes[13]. Similarly various coumarin chalcones in the solvent free media exhibit high potency as antibacterial agent[3].Introduction of fluoro and sulfonamide moieties into coumarin side chain hoping for an improvement of biological activity because incorporation of fluorine to various heterocycles is known to influence the biological activity[14] and the sulfonamide moiety itself possesses important antibacterial[15], anti-inflammatory[16], and antitumor activity[17]. Specifically 1,5 substituted benzothiazepine[18] are well known compounds for diverse therapeutically properties like antimicrobial[19], antihypertensive[20], calcium channel blocker[21], blood platelet aggregation inhibitory[22] and coronary vasodilatory effects[23]. Furthermore isoxazoline derivative of chalcones and coumarin possesses antibacterial activity against bacteria (gram+ve) and (gram-ve) and antifungal activity[24] and also 3-bromoacetyl coumarin with thiazo group (Schiff bases) possess a broad spectrum of biological importance[25].

Table no 1: Pharmacological activities of Coumarins

Sr. No.	Authors	Structure	Pharmacological Activity	Catalyst
1	Khadijah M. Al-Zaydi et. al. 2003 Ref.No.[13]		Pharmaceutical and Photochromic dyes	Solvent less dimethyl formamide dimethyl acetal (DMFDMA)
2	Khadijah M. Al-Zaydi et. al. 2003 Ref.No.[13]		Pharmaceutical and Photochromic dyes	Solvent less dimethyl formamide dimethyl acetal (DMFDMA)
3	Olayinka O. Ajani et. al. 2010 Ref.No.[3]		Anti bacterial Activity	Solvent less piperidine catalyst
4	Olayinka O. Ajani et. al. 2010 Ref.No.[3]		Anti bacterial Activity	Solvent less piperidine catalyst
5	Olayinka O. Ajani et.al. 2010 Ref.No.[3]		Anti bacterial Activity	Solvent less piperidine catalyst

6	Olayinka O. Ajani et. al. 2010 Ref.No.[3]		Anti bacterial Activity	Solvent less piperidine catalyst
7	Hua Zuo et. al. 2008 Ref.No.[14]		Antibacterial anti-inflammatory and anti tumor activity	Piperidine ethan
8	Jignesh P. Raval et al,2008 Ref.No.[1]		Anti microbial anti HIV, anti coagulant, anti allergenic activity	CH_3OH/2%NaOH
9	JigneshP. Raval et al,2008 Ref.No.[1]		Anti microbial anti HIV, anti coagulant, anti allergenic activity	CH3OH / Glacial CH3COOH
10	Hector Aguilar et. al. 2011 Ref.No.[30]	 And $CH_3COCH_2COOR_2$	–	Bi(NO3)3 5H2O
11	Milan Mladenovic et. al. 2009 Ref.No.[31]		Antimicrobial activity	Ammonium acetate
12	Milan Mladenovic et. al. 2009 Ref.No.[31]		Antimicrobial activity	P –toluene sulfonic acid in toluene

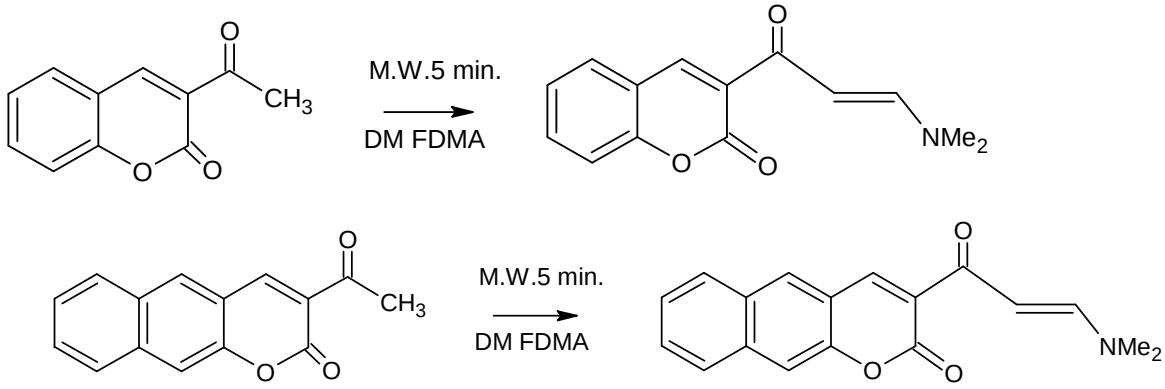
In the same the presence of a quinoline nucleus in the frame work of various pharmacologically active compound and they are valuables synthons used for the preparation of nano and meso structures with enhanced electronic and photonic properties[26]. The coumarins containing a Schiff base are expected to have enhanced antitumor and other biological activity. It is well established that the biological activity associated with the hydrazone compounds attributed to the presence of the active pharmacophore (-CONH-N=C-). Hence many hydrazone compounds containing this active moiety showed good anticancer bioactivities[27]. Various analogues of 4-substituted coumarin such as 4-chlorocoumarins exhibit antimicrobial activity,[28] Furocoumarins have cytotoxic activity devoid of

serious side effects[29].Apart from the above work, the present review also addresses the potential roles of coumarin substituted compounds and synthesis of coumarin derivatives using microwave irradiation in comparison with conventional methods of synthesis.

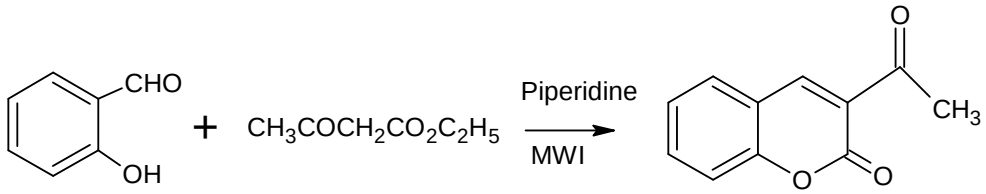
EXPERIMENTAL SECTION

Microwave Synthesis:

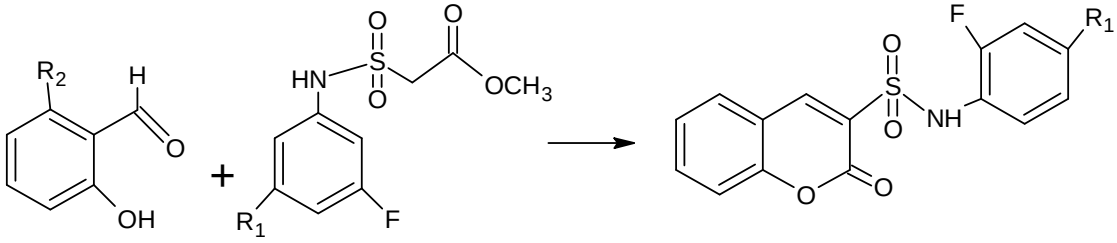
Rapid solventless synthesis of 3substituted coumarins and benzocoumarins by microwave irradiation: The utility of microwave in heterocyclic synthesis is now receiving considerable attention enaminones were smoothly obtained by reacting compounds 3-acetylcoumarin with dimethyl formamide dimethyl acetal (DMFDMA) in a domestic microwave for a very short time the yield being much higher than that obtained by conventional heating with a solvent[13].



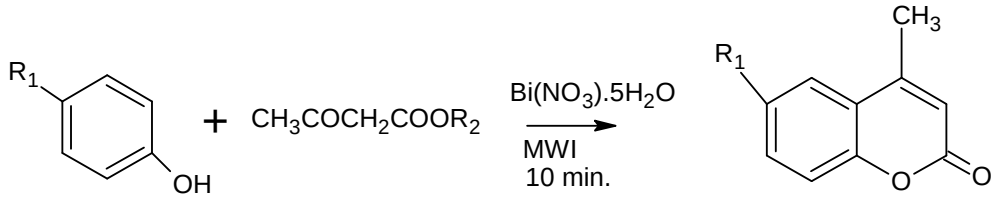
MW assisted synthesis of 3-acetylcoumarins: 3-acetyl coumarin was synthesised by the reaction of salisaldehyde with ethyl acetoacetate in the presence of catalytic amount of piperidine. On the basis of the experimental data from various studies that microwave enhanced chemical reaction rate can be faster than those of conventional heating methods by as much as a 1000 folds[3].



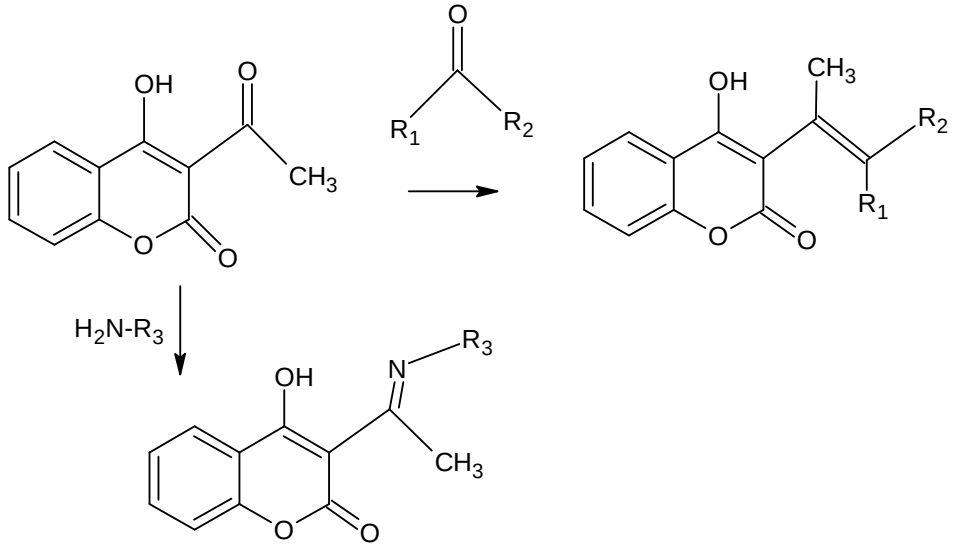
From Knoevenagel condensation: The Knoevenagel condensation of methyl 2- chlorosulfonyl acetate with subsituted salicylaldehydes led to N – fluoroaryl coumarin-3-sulfonamides[14].



MW assisted synthesis by Pechman reaction: Pechman reaction is the most important reaction for the synthesis of coumarin. Acidic reagents have been used to perform the cyclization reaction between phenol and an active dicarbonyl compound. The reaction of phenol and ethyl – acetoacetate in the presence of 5 mol% bismuth nitrate in the absence of solvent was performed in a domestic microwave oven for 8-10 minutes. This reaction produced coumarin in 80-85% yields[30].



Condensation of 4-hydroxy chromene-2-one by Knoevenagel condensation: The structural variety in the coumarins is achieved by the condensation of 4- hydroxy chromene-2- one with different carboxyl, ester and cyano derivatives. This is synthesized by knoevenagel condensation both conventional and fast microwaves proceduce coumarln schiff- base derivatives are also synthesized by the method of synthesis[31].



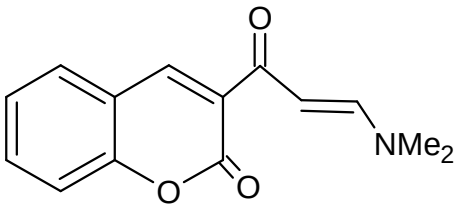
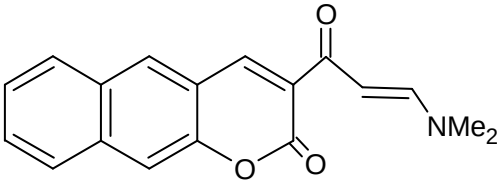
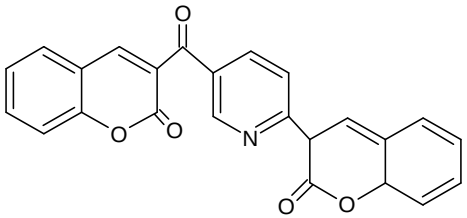
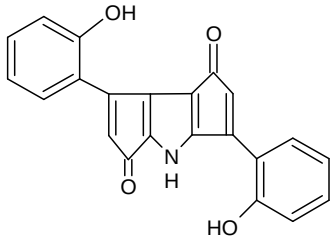
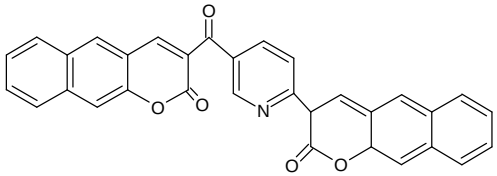
Natural coumarins possessing anti-HIV activity

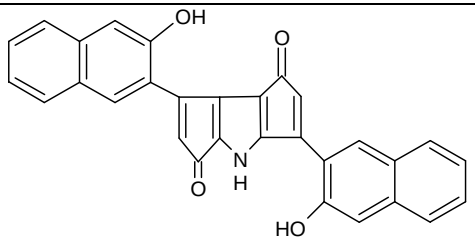
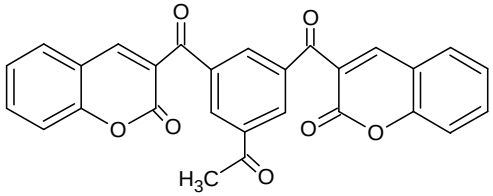
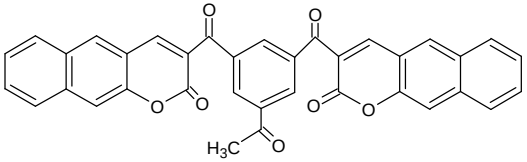
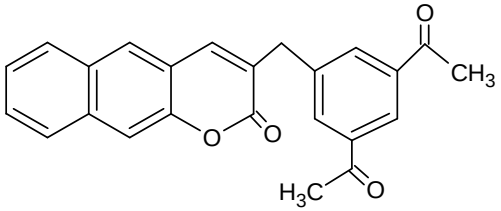
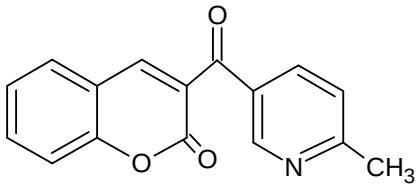
Dipyranocoumarins-Calanolides:(+)-Calanolide A, (+)-[10R, 11S, 12S]-10, 11-trans-dihydro-12-hydroxy-6,6,10,11-tetramethyl-4-propyl-2H, 6H-benzo[1,2-b:3,4-b':5,6b"] tripyran-2-one, is a novel nonnucleoside RT inhibitor (NNRTI) with potent activity against HIV-1.

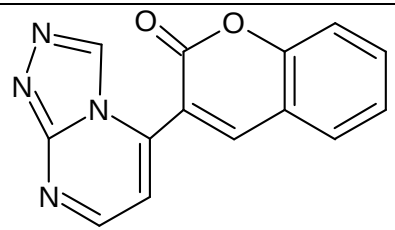
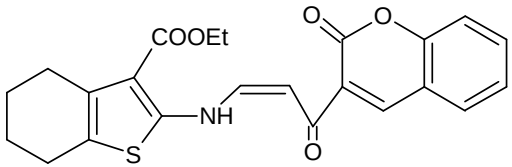
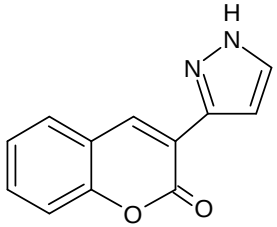
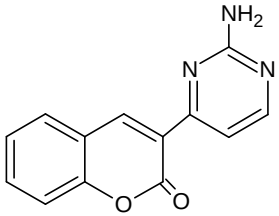
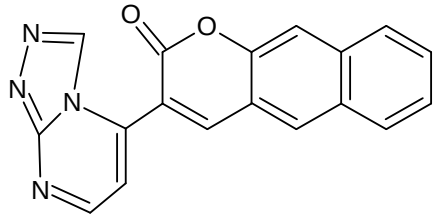
Costatolides: Two isomers of calanolide A, (-)-calanolide B (costatolide) and (-)-dihydrocalanolide B (dihydrocostatolide), possess antiviral properties similar to those of calanolide A. Each of these three compounds has properties of NNRTIs. The calanolide analogues, however, exhibit enhanced antiviral activity against drug-resistant viruses after NNRTI treatment. Costatolide and dihydrocostatolide are highly effective inhibitors of clinical strains, including those representing various HIV-1 clades, Sis, NSIs, T-and M-tropic isolates.

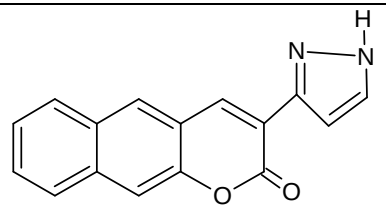
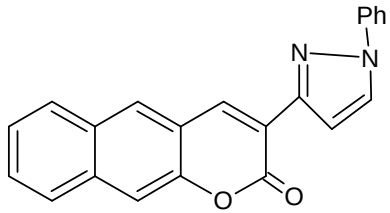
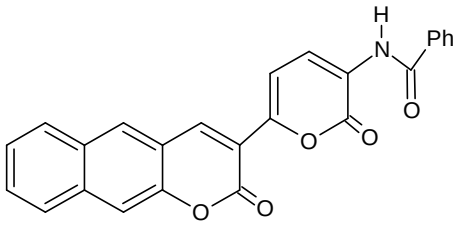
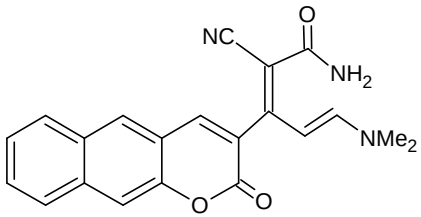
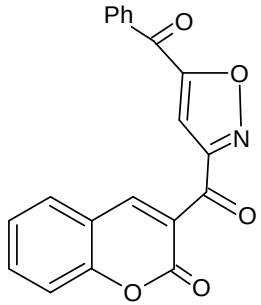
Inophyllums: The seeds of Calophyllum cerasiferum vesque (family-*clusiaceae*) and calophyllum inophyllum linn.(family-*clusiaceae*) contain several known coumarins, among them the potent HIV-1 RT inhibitors costatolide and inophyllum P. calophyllum cerasiferum contained (-)-calanolide B[2].

Table No.2:Comparison between microwave-assisted and conventional method of synthesis in terms of yield and time [13]

Sr. No.	Structure	Microwave Irradiation		Conventional Heating		Ref. No.
		Time (min)	Yield (%)	Time (hr.)	Yield (%)	
1		5	96	6	75	32
2		5	95	6	73	32
3		2	55	½	65	32
4		5	85	_	_	32
5		_	_	2	64	32

6		3	96	_	_	32
7		_	_	2	66	33
8		_	_	2	49	33
9		_	_	2	45	33
10		2	45	_	_	32

11		5	65	6	70	32
12		5	69	2	45	32
13		5	60	4	60	32
14		25	60	3	45	32
15		5	90	2	74	32

16		5	69	4	65	32
17		5	61	4	59	32
18		5	90	1	60	32
19		1	60	2	45	32
20		10	54	24	41	34

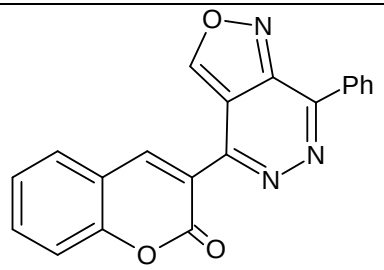
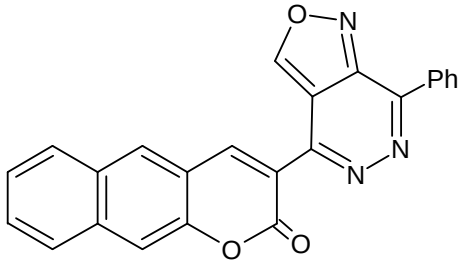
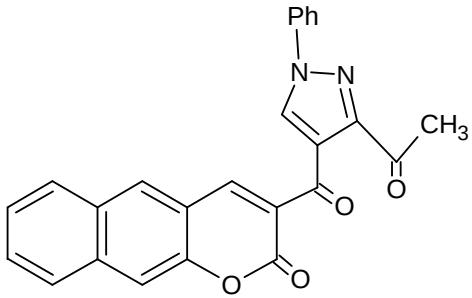
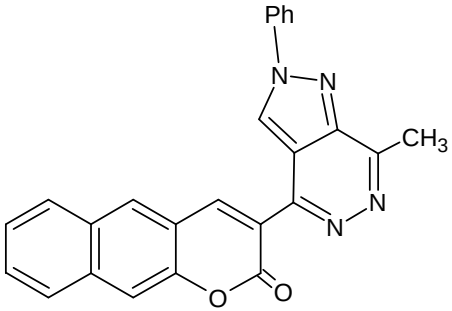
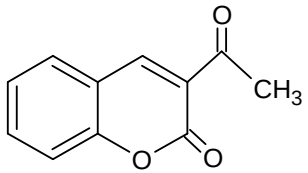
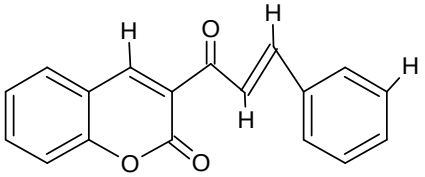
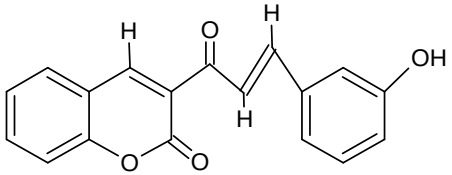
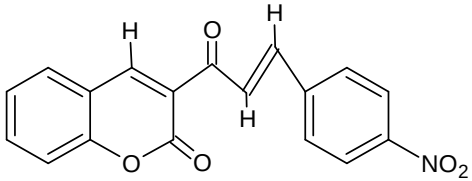
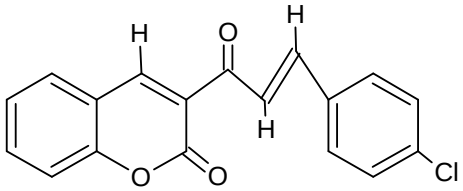
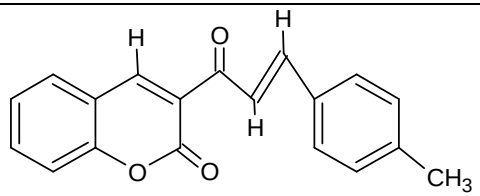
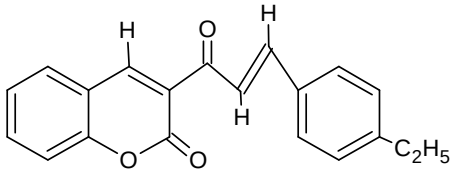
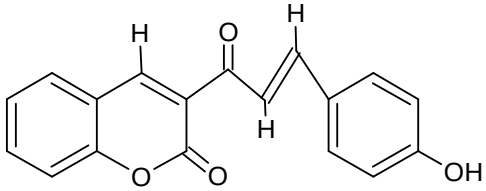
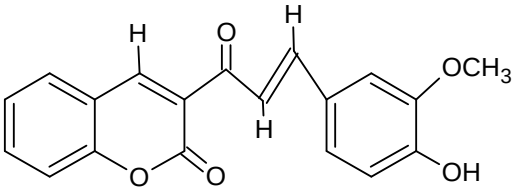
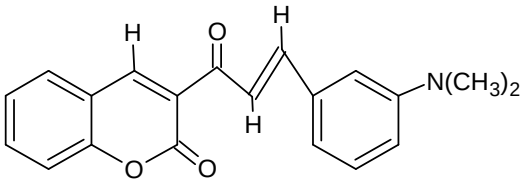
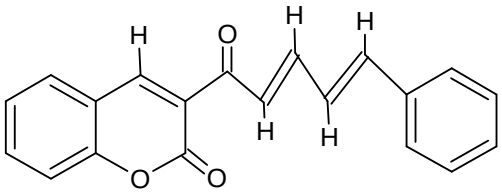
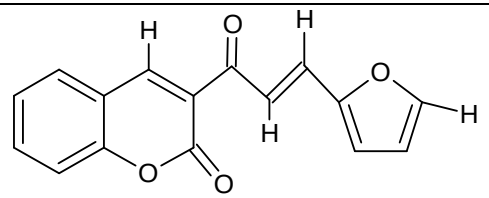
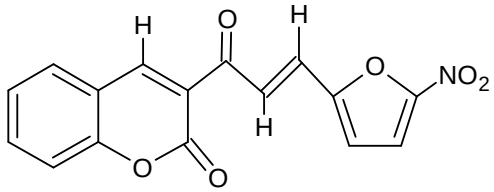
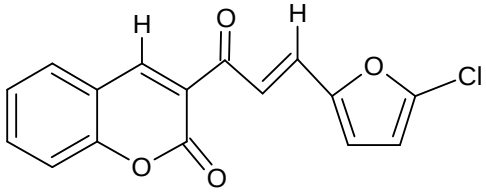
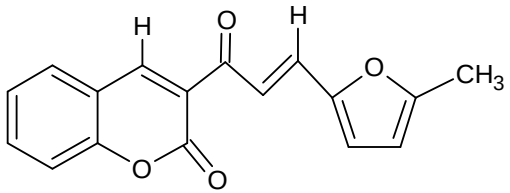
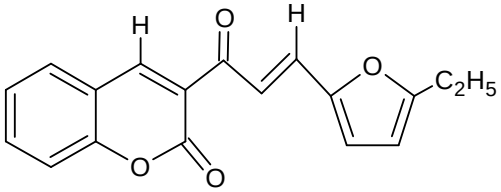
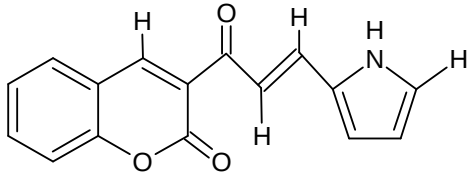
21		_	_	371		34
22		10	54	24	45	34
23		_	_	3	74	34
24		10	55	24	43	34

Table No.3: Comparison between microwave-assisted and conventional method of synthesis in terms of yield and time[3]

Sr. No.	Structure	Microwave Irradiation		Conventional Heating		Ref. No.
		Time (min)	Yield (%)	Time (hr.)	Yield (%)	
1		1	92.6	_	_	35
2		1-3	97	_	_	35
3		1-3	89.1	_	_	35
4		1-3	78.3	_	_	35
5		1-3	77.1	_	_	35

6		1-3	92.5	_	_	35
7		1-3	95.1	_	_	35
8		1-3	97.8	_	_	35
9		1-3	77.1	_	_	35
10		1-3	81.6	_	_	35
11		1-3	73.3	_	_	35

12		1-3	87.2	_	_	35
13		1-3	66.8	_	_	35
14		1-3	59.2	_	_	35
15		1-3	74.8	_	_	35
16		1-3	66.5	_	_	35
17		1-3	71.4	_	_	35

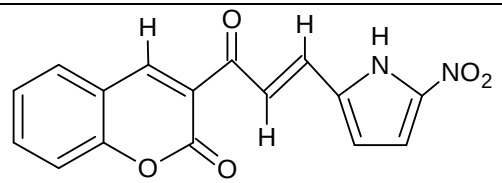
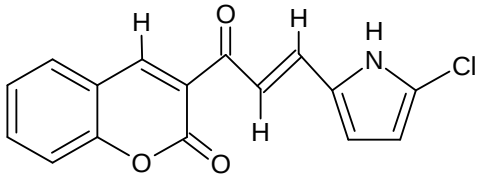
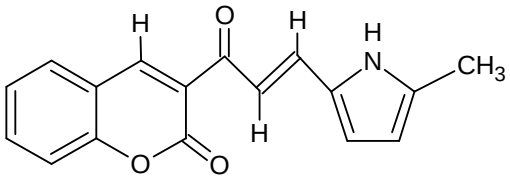
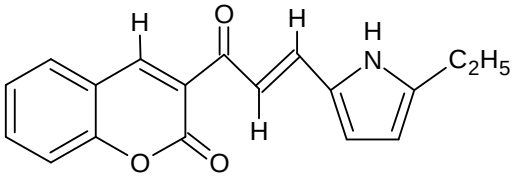
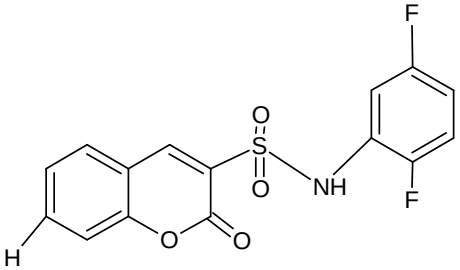
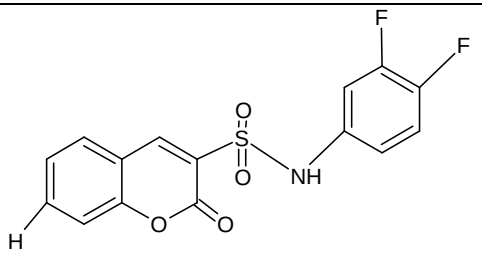
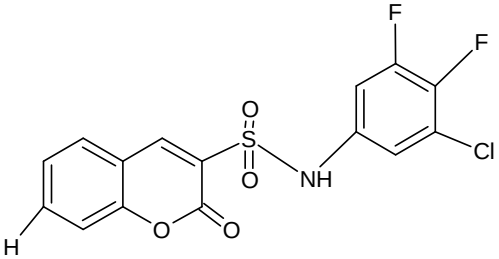
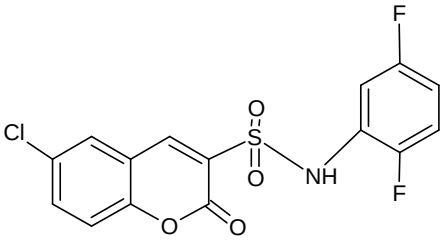
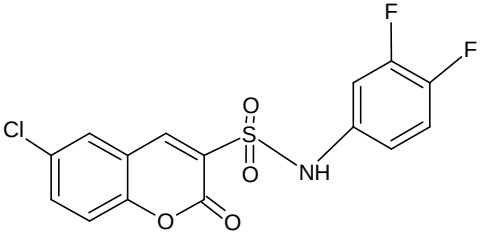
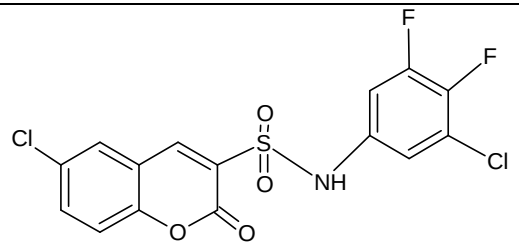
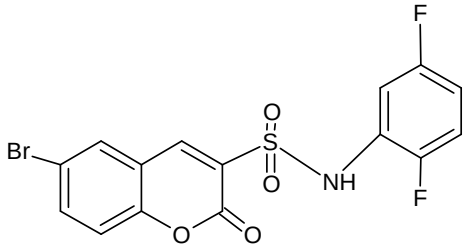
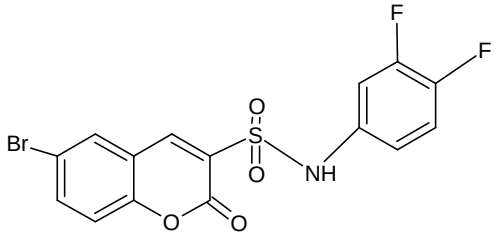
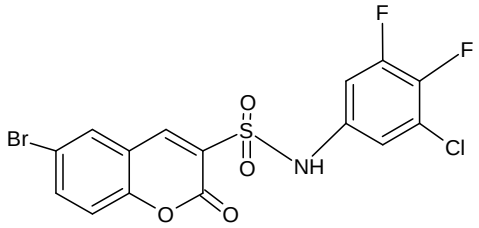
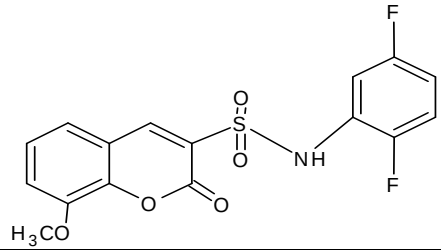
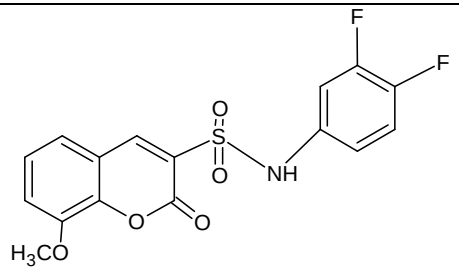
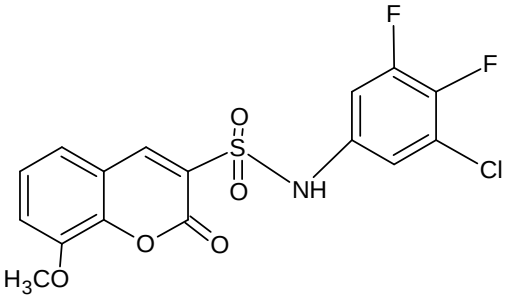
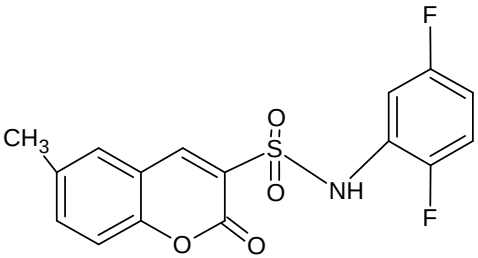
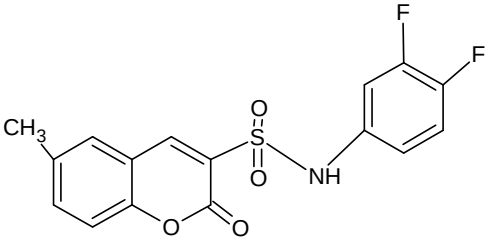
18		1-3	63.7	_	_	35
19		1-3	55.8	_	_	35
20		1-3	50.1	_	_	35
21		1-3	50.8	_	_	35

Table No.4:Comparison between microwave-assisted and conventional method of synthesis in terms of yield and time[14]

Sr. No.	Structure	Microwave Irradiation		Conventional Heating		Ref. No.
		Time (min)	Yield (%)	Time (hr.)	Yield (%)	
1		5	84	8	78	36

2		5	86	8	80	36
3		4	76	8	71	36
4		8	85	8	78	36
5		8	86	8	78	36

6		8	88	8	84	36
7		10	84	8	82	36
8		8	86	10	80	36
9		10	85	10	78	36
10		10	92	10	85	36

11		10	90	10	86	36
12		8	86	10	82	36
13		12	74	10	70	36
14		12	84	10	79	36

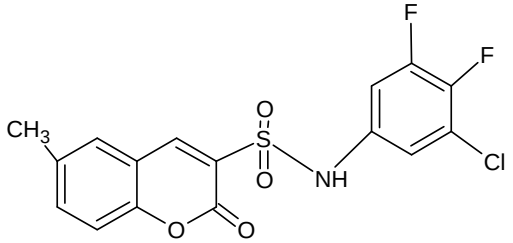
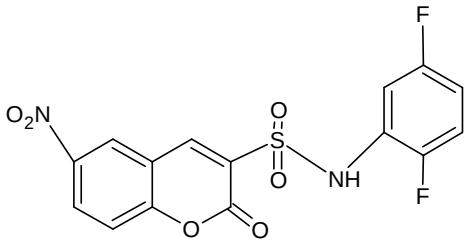
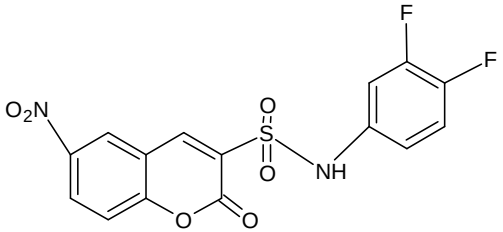
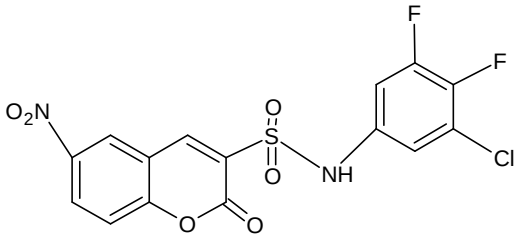
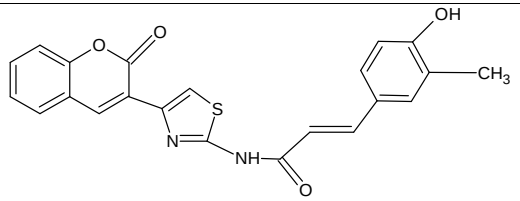
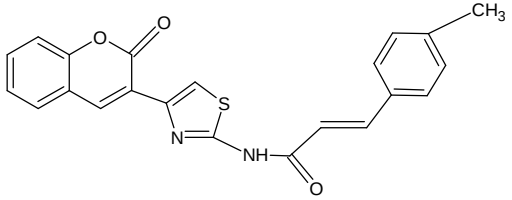
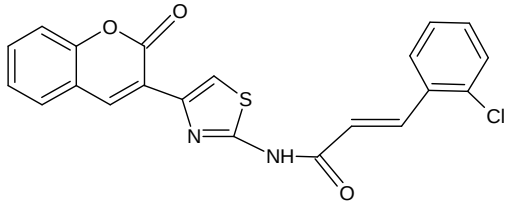
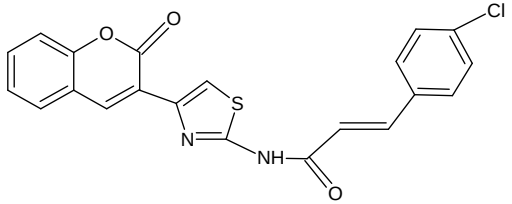
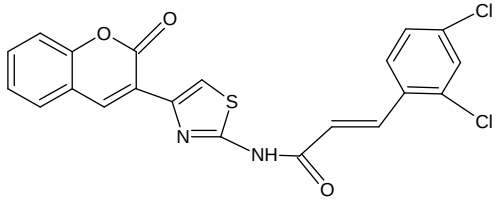
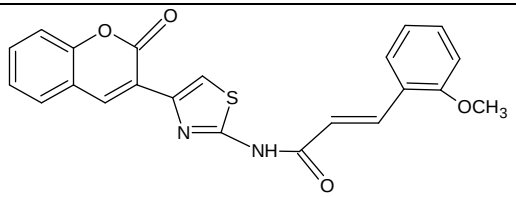
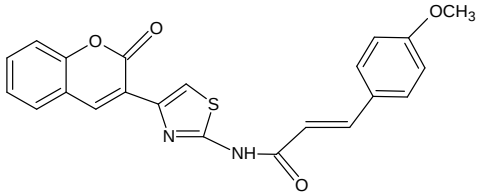
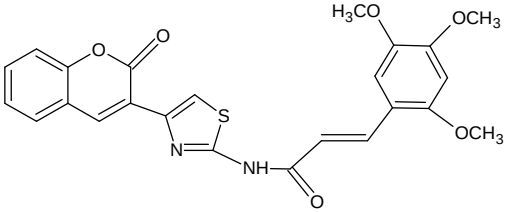
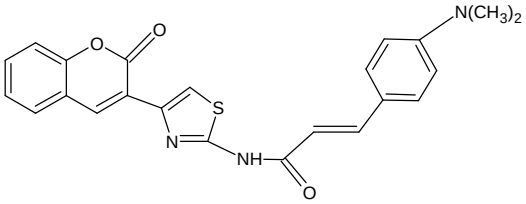
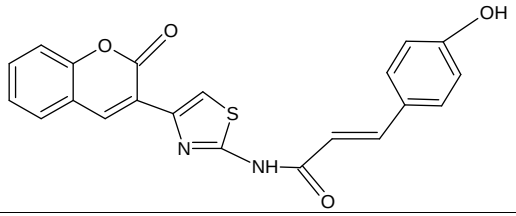
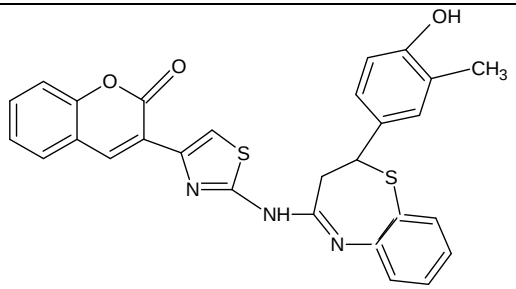
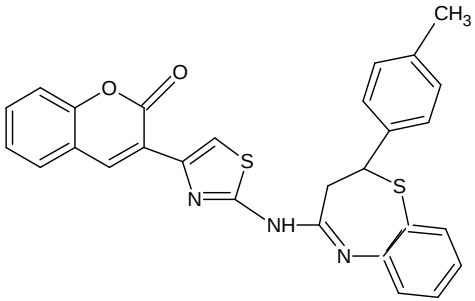
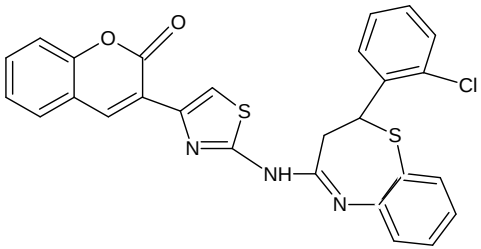
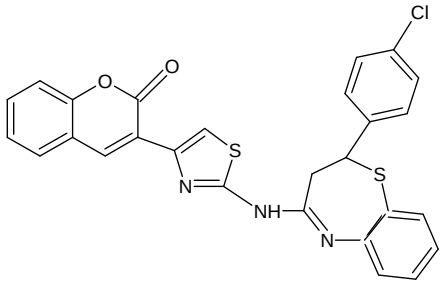
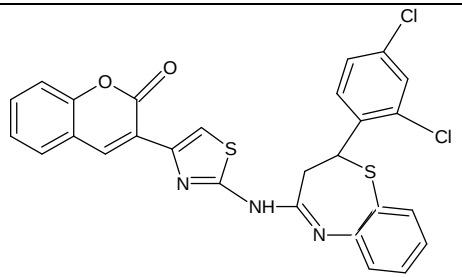
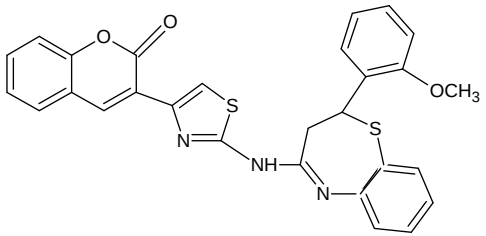
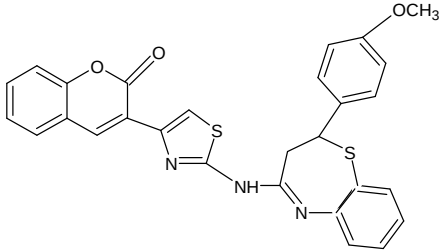
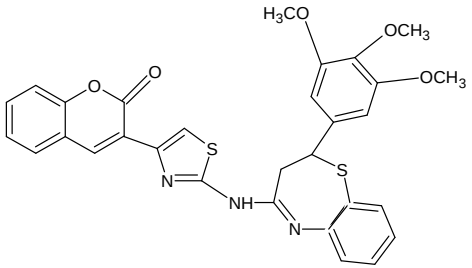
15		12	80	10	77	36
16		12	82	10	78	36
17		12	80	10	71	36
18		12	78	10	72	36

Table No.5:Comparison between microwave-assisted and conventional method of synthesis in terms of yield and time [18]

Sr. No.	Structure	Microwave Irradiation		Conventional Heating		Ref. No.
		Time (min)	Yield(%)	Time (hr.)	Yield (%)	
1		3.5	82	6	61	37, 38
2		3.0	86	6	58	37, 38
3		3.5	84	6.5	63	37, 38
4		3.5	87	7	66	37, 38
5		3.5	88	7	67	37, 38
						37, 38

6		3.5	89	7	68	
7		3	81	8	69	37, 38
8		3.5	86	8	70	37, 38
9		3.0	88	6.5	71	37, 38
10		3.5	87	6	72	37, 38

11		3.5	88	7	73	37, 38
12		3.0	92	7	77	37, 38
13		3.0	91	8	71	37, 38
14		3.5	92	8	72	37, 38

15		3.5	88	6	68	37, 38
16		3.5	85	7	69	37, 38
17		3.5	87	9	71	37, 38
18		3.5	88	9	66	37, 38

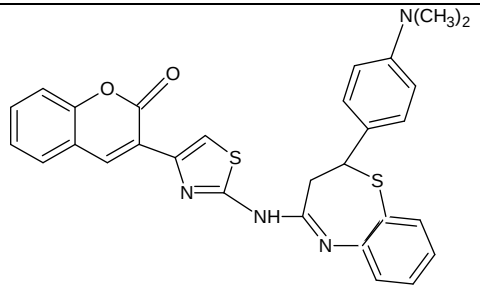
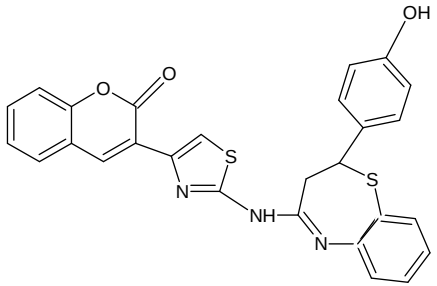
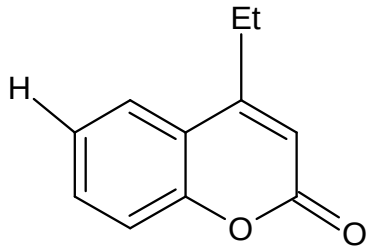
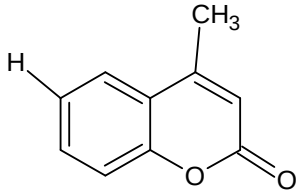
19		3.5	89	8	65	37, 38
20		3.5	92	8	72	37, 38

Table No.6:Comparison between microwave-assisted and conventional method of synthesis in terms of yield and time [30]

Sr. No.	Structure	Microwave Irradiation		Conventional Heating		Ref. No.
		Time (min)	Yield (%)	Time (hr.)	Yield (%)	
1		8-10	80-85	1-3	_	39, 40, 41
2		8-10	80-85	1-3	_	39, 40, 41

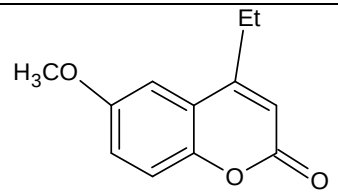
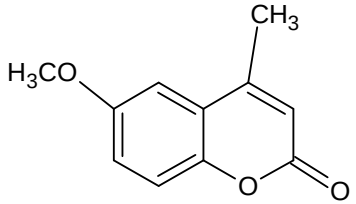
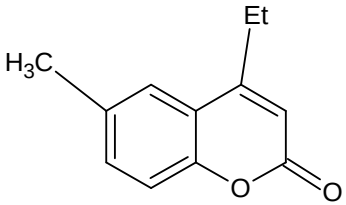
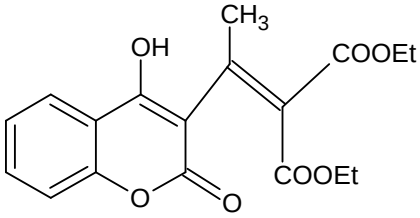
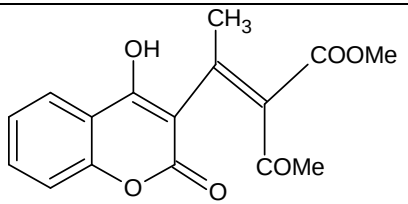
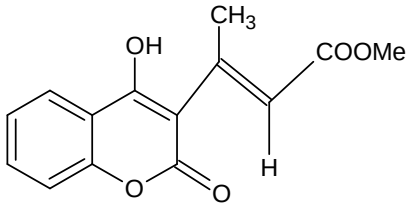
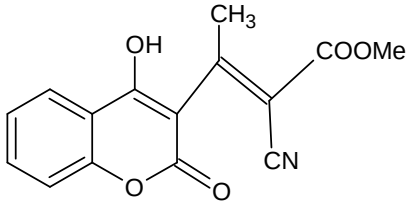
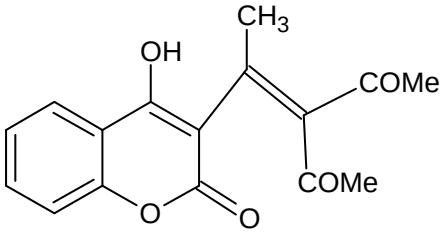
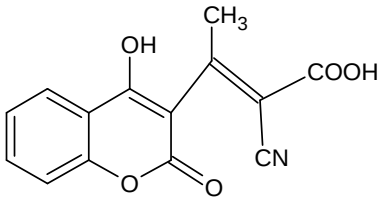
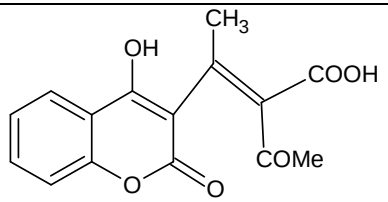
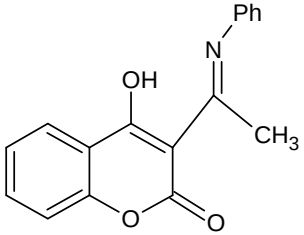
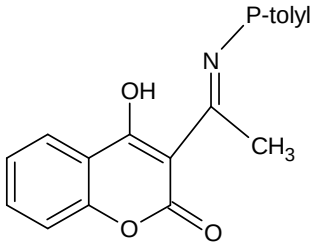
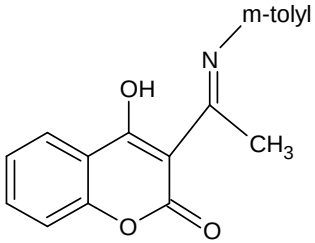
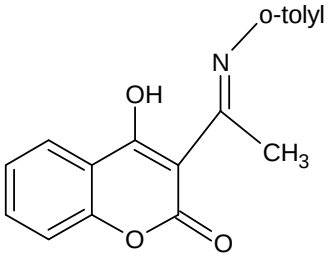
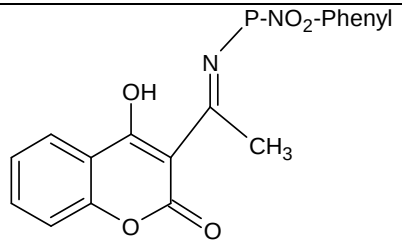
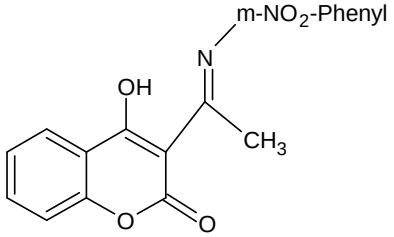
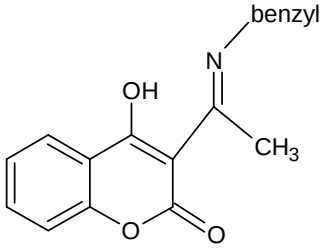
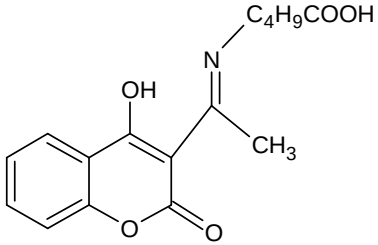
3		8-10	80-85	1-3	_	39, 40, 41
4		8-10	80-85	1-3	_	39, 40, 41
5		8-10	80-85	1-3	_	39, 40, 41

Table No.7:Comparison between microwave-assisted and conventional method of synthesis in terms of yield and time [31]

Sr. No.	**Structure**	Microwave Irradiation		Conventional Heating		Ref. No.
		Time (min)	Yield (%)	Time (hr.)	Yield (%)	
1		7	96	8	_	42

2		7	97	8	_	42
3		9	96	8	_	42
4		7	94	8	_	42
5		10	94	8	_	42
6		6	84	8	_	42

7		5	87	8	_	42
8		3	95	10-12	_	42
9		3	97	10-12	_	42
10		3	94	10-12	_	42
11		3	94	10-12	_	42

12		3	92	10-12	_	42
13		3	97	10-12	_	42
14		3	97	10-12	_	42
15		3	87	10-12	_	42

CONCLUSION

Coumarin and coumarin-related compounds have proved for many years to have significant therapeutic potential. They come from a wide variety of natural sources and new coumarin derivatives are being discovered or synthesized on a regular basis. Coumarin is a simple molecule and many of its derivatives have been known for more than a century. However, their vital role in plant and animal biology has not been fully exploited. It is evident from the research described that coumarin and coumarin-related compounds are a plentiful source of potential drugs candidate in relation to its safety and efficacy.New coumarin derivatives have been synthesized using conventional and microwave heating mathodology and characterized. The advantages in the use of microwave methodology are shorter reaction times, higher yields and simplified work up procedures for the point of purification of the prepared compound.The combination of solvent free reaction condition and microwave irradiation leads to significantly reduced reaction times, enhanced conversions and sometime higher selectivity with several advantages for the eco-friendly approach, termed as “Green Chemistry”.

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