

A Green Method Synthesis and Antimicrobial Activity of 2-Amino-4H-Chromene Derivatives

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Agro-waste stuff ash extracted solution have been emerging in recent practicing green reaction catalysis in organic synthesis. The derived media have been demonstrated in reactions like Sonogashira, Dakin, Henry, Suzuki-Miyaura, amide bond formation. Traditionally, these reactions were performed in organic solvents and used expensive catalysts with elevated temperature. These catalytic media showed distinct properties during reaction course such as, *in situ* base, homogeneous catalysts and reducing agent. The present work reviewed green protocol synthesis of 2-amino-4*H*-chromene derivatives employing reaction of substituted benzaldehyde, malononitrile and α/β -naphthol or resorcinol; three component reaction catalyzed in the presence of water extract of banana peel ash (WEB) extraction under microwave and grindstone methods is described. The reaction found eco-friendly, simple reaction condition with easy separation of final product in pure form. The final product was characterized for its homogeneity using spectroscopic techniques. Some of the selected chromene derivatives (**3b**, **3d**, **3g**) prepared in this method is tested for its *in vitro* antimicrobial studies. The bioassay revealed that products **3b** and **3d** showed positive response and comparable antimicrobial activity with the reference compounds.

Keywords: Chromene, Green method, Water extract of banana peels, Microwave irradiation, Antimicrobial activity.

INTRODUCTION

Green chemistry in the past two decades playing important environmental impact, and continuous effort to come up with an alternative methodology that minimizes use of the hazardous solvents during chemical synthesis and reduction of reaction time [1]. Therefore in the recent year's use of agro-waste stuff ash extract as a chemical surrogates becomes one of the most exciting discoveries of organic chemists [2]. Many authors reported a novel route for the Suzuki-Miyaura cross-coupling reactions using water extract of banana peel ash (WEB) as solvent system as well as *in situ* base for the reaction [3-5]. It is also well documented that burnt ash contains K, Na, CO₃²⁻ and Cl⁻ as a major constituents along with a trace elements like Ca, Mg and Cu [6]. It is believed that presence of carbonates of Na and K of banana peel ash extract act as a base and promotes reactions in the faster rate with microwave irradiation. Thus, the feasibility of using inherent basicity of water extract of banana peel ash in this reaction required serious consideration. In medicinal chemistry, 2-amino-4H-chromene derivative scaffolds plays an important role with wide range of biological applications, such as anticancer, antiallergic, antiproliferative, antiviral, antibacterial and apoptosis inducers [7-9]. Such wide range of biological applications of chromene derivatives made resear-

cher to discover novel and competitive methodologies for the synthesis, where the derivative can be synthesized one pot with an environmentally friendly protocol are of recent interest of synthetic chemists [10-13]. Multicomponent reactions have been employed successfully to generate highly diversified combinatorial libraries for high through put screening of biomolecules [14,15]. This type of reaction was highly important in medicinal chemistry because it allows highly complex poly functional molecule through one-pot simple and one step reaction [16]. Multicomponent reactions with environmentally benign solvents and catalytic synthesis are one of the suitable green protocols, which meet the requirements of green aspects of developing library of biomolecules by organic synthesis [17]. Organic reaction carried out in water has become highly popular in recent trends due to its specific medicating organic reactions and it's environmentally friendly [18]. Several procedures for the synthesis of chromene derivatives through multicomponent reactions are reported [19]. Chromenes are generally synthesized through three component reaction of phenol/naphthol, aromatic aldehyde and malononitrile in the presence of catalyst [20,21]. A vast variety of catalyst have been reported by several authors such as HTMAB [22], DABCO [23,24], p-TSA [10,25], CTA-Cl [26], [bimim]-OH [27], thiourea oxide, TBA-Br [28], MgO nanoparticles [29], STA [30], CuSO₄·5H₂O [31], ammonium acetate [32], Et₃N [33], K₂CO₃ [34], Fe(HSO₄)₃ [35] and piperidine [36]. Most of these traditional reported methods required purification steps, long reaction time and use of volatile organic solvents with elevated temperatures [26,37]. Microwave accelerated organic reactions have been received great importance, due to their simplicity in operations, elimination of the solvents and reduction of the reaction time resulting high yield with purity compare to conventional heating [20,38,39]. The application of microwave accelerated has been employed in various useful transformations involving condition, oxidation [40-43], reduction [44], protection-deprotection [45-49], rearrangement reactions synthesis of different heterocyclic scaffolds [50]. As part of our research interested on green method synthesis of biologically active heterocyclic scaffolds, we present one pot product formation of 2-amino-4H-chromene in water extract of banana peel ash [3,4]. The synthesis is influenced in the presence of microwave accelerated and grindstone method.

EXPERIMENTAL

All the reagents used in the synthesis were purchased from Sd-fine Chemicals and were used without further purification. The melting points were determined by the open capillary tube method and are uncorrected. The water extract of banana peel ash (WEB) solution is subjected to elemental analysis by the ESICO Microprocessor flame photometry model 1382. FT-IR spectra were measured with Thermo Fischer scientific using KBr pellet method, ¹H-, ¹³C-NMR spectra were obtained in DMSO- d_6 on Agilent spectrometer at 400 MHz. The chemical shifts are reported in ppm (δ) relative to tetramethylsilane as internal standard. The mass spectra are recorded in Waters Synapt G2, USA.

Preparation of water extract of banana peel ash (WEB): The WEB extraction is done by reported procedure using banana peels collected from local area (*Musa acuminata*) [3,5]

General procedure for the synthesis of 2-amino-4H-chromenes

Method 1: In a clean mortar the corresponding aldehyde (2 mmol), malononitrile (2 mmol), resorcinol/ α -naphthol or β -naphthol and 3 mL of WEB solution taken. The mixture ground for 15-25 min, the progress of the reaction was monitored by TLC. After completion of the reaction, added 5 mL of distilled water and filtered, the residue is washed with excesses amount of distilled water followed by cold methanol. The crude product was recrystallized by alcohol and characterized by FT-IR, ¹H-, ¹³C NMR using DMSO-*d*₆ solvent and HR-MS.

Method 2: In a clean round bottomed flask, the corresponding aldehyde (2 mmol), malononitrile (2 mmol), resorcinol/ α -naphthol or β -naphthol and 3 mL of WEB solution taken, the mixture is subjected to microwave irradiation at 300 W power for 3 min and the progress of the reaction was monitored by TLC. After completion of the reaction, added 5 mL of distilled water and filtered, the residue is washed with excesses amount of distilled water followed by cold methanol. The crude product was recrystallized by alcohol and characterized by FT-IR, ¹H-, ¹³C-NMR in DMSO-*d*₆ solvent and mass by HR-MS.

Spectral data of selected compounds

Compound 3a: m.p. 232-234 °C, IR (KBr, v_{max}, cm⁻¹): 3497.61 (-OH), 3428.98 (NH₂), 2928.67 (C-H), 2192.28 (C=N),

1647.56 (C=C), 1449.69, 1408.18, 1313.09, 1249.82, 1053, ¹H NMR (DMSO- d_6 400 MHz, δ ppm): 4.62 (s,1H), 6.47 (t, 2H), 6.82 (m, 3H), 7.18 (m, 3H), 7.26 (t, 2H), 9.6 (s, 1H); ¹³C NMR (DMSO- d_6 400 MHz): 102.12, 112.32, 113.70, 120.57, 126.56, 127.31, 129.83, 146.30, 148.81, 157.02, 160.19. HR-MS theoretical [M]⁺264, observed *m*/*z* 265.01. Elemental analysis of C₁₆H₁₂N₂O₂ Calc. (found) %: C 72.72 (72.64), H 4.58 (4.59), N 10.60 (10.58) and O 12.11 (12.13).

Compound 3f: m.p. 186-188 °C, IR (KBr, v_{max} , cm⁻¹): 3421.33 and 3360.02 (NH₂), 3310.02 (C-H), 2190.11 (C=N), 1592.11 (C=C), 1588.12, 1499.54, 1488.35, 1445.34, 1187.11. ¹H NMR (DMSO-*d*₆ 400 MHz, δ ppm): 2.22 (s, 3 H), 4.34 (s, 1H), 6.02 (d, 1H), 6.41 (d, 1H), 6.59 (d, 1H), 6.79 (brs, 2H), 7.33 (d, 1H), 7.45 (d, 1H); ¹³C NMR (DMSO-*d*₆, 400 MHz): 13.98, 44.39, 101.28, 112.32, 120.19, 121.85, 122.56, 120.72, 129.93, 142.11, 142.28, 142.55, 160.01, 160.97. HR-MS theoretical [M]⁺278.00, observed *m*/*z* 278.45. Elemental analysis of C₁₇H₁₄N₂O₂ Calc. (found) %: C 73.37 (73.38), H 5.07 (4.09), N 10.07 (10.01) and O 11.50 (11.44).

Compound 3j: m.p.194-196 °C, IR (KBr, v_{max} , cm⁻¹): 3467.23 and 3339.56 (NH₂), 3126.33 (C-H), 2189.45 (C=N), 1499.24, 1478.22, 1230.22, 1168.45. ¹H NMR (DMSO-*d*₆ 400 MHz): 3.98 (s, 3H), 4.22 (s, 3H), 4.40 (s, 1H), 5.34 (s, 2H), 7.45-7.85 (m, 9H); ¹³C NMR (DMSO-*d*₆, 400 MHz): 15.89, 44.48, 111.22, 113.29, 119.22, 123.56, 126.93, 129.68, 130.34, 142.76, 142.2, 145.22, 158.89,164.56. HR-MS theoretical [M]⁺358, observed *m*/*z* 358.22. Elemental analysis of C₂₂H₁₈N₂O₃ Calc. (found) %: C 73.32 (73.29), H 5.59 (5.55), N 7.77 (8.01) and O 13.32 (13.36).

Compound 3n: m.p. 238-240 °C, IR (KBr, v_{max} , cm⁻¹): 3260.01 and 3440.34 (NH₂), 3123.89 (C-H), 2197.32 (C=N) 1640.11, 1496.90 (C=C), 1491.23, 1402.13, 1229.23, 1154.09, 1051.33, ¹H NMR (DMSO-*d*₆ 400 MHz, δ ppm): 8.34 (d, 1H), 7.76 (d, 1H), 7.53 (m, 3H), 7.49 (m, 2H), 7.29 (m, 3H), 7.04 (s, 2H), 4.75 (s, 1H); ¹³C NMR (DMSO-d6, 400 MHz): 41.22, 51.86, 112.34, 120.34, 121.67, 123.54, 124.31, 125.05, 126.87, 127.22, 128.33, 128.66, 131.90, 139.25, 144.53, 150.83, 160.23. HR-MS theoretical [M]⁺376, observed *m/z* 376.61. Elemental analysis of C₂₀H₁₃N₂OBr Calc. (found) %: C 63.34 (63.30), H 3.99 (3.98), N 7.39 (7.38), O 4.22 (4.21) and Br 21.07 (21.02).

Compound 3o: m.p. 224-226 °C, IR (KBr, v_{max} , cm⁻¹): 3350.25 and 3289.98 (NH₂), 3034.62 (C-H), 2189.54 (C=N), 1495.33, 1498.22, (C=C), 1521.20, 1323.11, 1241.44, 1129.02, 1112.33, ¹H NMR (DMSO-*d*₆, 400 MHz, δ ppm): 8.23 (d, 1H), 7.54 (t, 2H), 7.80 (m, 3H), 7.43 (d, 2H), 7.23 (d, 2H), 6.89 (d, 1H), 6.77 (s, 2H), 4.79 (s, 1H), ¹³C NMR (DMSO-*d*₆, 400 MHz): 43.34, 122.66, 122.82, 125.41, 126.98, 128.92, 129.34, 130.43, 130.91, 132.23, 132.44, 133.56, 134.02, 134.98, 137.54, 145.67, 147.22, 162.21. HR-MS theoretical [M]⁺315, observed *m/z* 315.67. Elemental analysis of C₂₀H₁₃N₂OF Calc. (found) %: C 75.46 (75.51), H 4.75 (4.73), N 8.80 (8.84), O 5.03 (4.98) and F 5.97 (5.86).

Compound 3p: m.p. 241-243 °C, IR (KBr, v_{max} , cm⁻¹): 3389.01, 3334.40 (NH₂), 3116.24 (C-H), 2178.33 (C=N), 1589.06, 1469.98 (C=C), 1434.22, 1367.56, 1256.67, 1198.45, 1089.21, ¹H NMR (DMSO-*d*₆ 400 MHz, δ ppm): 3.34 (s, 3H), 3.65 (s, 3H), 5.30 (s, 3H), 7.00 (s, 2H), 7.22-7.76 (m, 9H); ¹³C NMR (DMSO-*d*₆, 400 MHz): 42.14, 121.26, 124.42, 124.81, 125.73, 127.89, 128.99, 130.22, 131.41, 131.68, 132.92, 133.32,

133.52, 134.78, 137.44, 138.23, 146.08, 162.44. HR-MS theoretical [M]⁺358, observed 359.44. Elemental analysis of $C_{22}H_{18}N_2O_3$ Calc. (found) %: C 73.32 (73.22), H 5.59 (5.48), N 7.77 (7.68) and O 13.32 (13.24).

RESULTS AND DISCUSSION

Herein, we wish to report green protocol efficient synthesis of 2-amino-4H-chromene derivatives (3a-p) catalyzed WEB under the influence of both microwave irradiation and grindstone methods. Konwar et al. [3] have reported the extraction of WEB from agro-waste of banana peels-ash and we adopted the same procedure for the extraction [3], but here we selected banana peels from local available with different variety (Musa acuminata). Musa species are grown in 135 countries primarily for the fruit and to a lesser extent to make fiber, banana wine and banana beer. Here we also analyzed the composition of our WEB using Microprocessor flame photometry model-1382, experimental data showed the presence of Li (55.2ppm), Na (15.4 ppm) and K (116.25 ppm). This study shows the presence of higher concentration of K, it is comparable with reported data. Surprisingly, our local variety WEB solution application explored in the cyclocondensation of three component reaction for chromene synthesis. The authors also developed and employed other greener reagents derived from agro-waste ash for the

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synthesis of chromene as well as other bioactive molecule synthesis [5,51]. Firstly, we started with the optimization of green catalyst for the model reaction compound 3a, initial with zero to the 5 mL with interval of 1 mL and found the minimum amount required 3 mL of WEB solution, gave best yield even after increase the volume. Therefore in the present work, we have used 3 mL of WEB for all our reactions. Surprisingly, the product formation not observed without WEB, this result revealed the importance of WEB media required for the reaction. Thus, reaction of three reactants in equimolar concentration in the presence of 3 mL of WEB at room temperature in grinding method about 15-25 min resulted the separation of solid product (Scheme-I). The reaction found to be simple and not required organic solvent for the reaction as well for the extraction. After reaction, the mixture is diluted with water, filtered and washed with cold methanol and water (1:1) resulted spectroscopically pure product 3a. Further the reaction is also extended under microwave irradiation at 300 W power with same equivalent reactants catalyzed by WEB solution, unexpectedly found the separation of product after 3 min irradiation. These product separated was found to be pure enough to do direct characterization without further purification. The homogeneity of the product **3a** was confirmed by FT-IR, ¹H-, ¹³C-NMR and mass spectrometry. The mechanism of the reaction is well documented



Scheme-I: General synthesis of 2-amino-4*H*-chromene

TABLE-1

PRODUCTS, YIELDS AND PHYSICAL CONSTANTS OF DIFFERENT ALDEHYDES, MALONONITRILE AND PHENOLS						
S. No.	Aldehyde	Phenol	Product	Time (min)	Yield (%)	m.p. (°C)
1	C ₆ H ₅ (1)	Resorcinol (2)	HO O NH ₂	2.5	78	232-234
2	4-Br-C ₆ H ₄	Resorcinol	(3a) Br HO HO NH ₂ (3b)	3.0	75	240-242





ANTIMICROBIAL ACTIVITIES OF SELECTED COMPOUNDS							
	Compounds -	Zone of inhibition (mm)					
	Compounds	75 μg	50 µg	25 µg	10 µg	5 µg	
Klebsiella	3b	13	10	R	R	R	
	3d	15	13	12	R	R	
	3g	12	10	R	R	R	
	Ciprofloxacin	-	-	-	30	-	
E. coli	3b	20	18	15	R	R	
	3d	25	23	20	10	R	
	3g	20	18	10	R	R	
	Ciprofloxacin	-	-	-	32	-	
A. niger	3b	25	20	18	10	R	
	3d	28	25	20	R	R	
	3g	10	08	R	R	R	
	Fluconazole	-	-	26	-	-	
Candida	3b	27	25	20	R	R	
	3d	28	25	24	20	18	
	3g	23	20	18	R	R	
	Fluconazole	_	-	24	-	-	

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in the literature for the reaction of benzaldehyde, malononitrile to give Knoevenagel product, subsequent Michael addition of resorcinol or α/β -naphthol to aldol product gives intermediate followed by cyclization of intermediate to give imine afford final product [38]. After optimizing the reaction conditions, different substituted aldehydes were examined, the feasibility of this product formation whose results are tabulated in Table-1. Further, resorcinol or α/β -naphthols reaction with malononitrile and substituted aldehyde resulted 2-amino-4*H*-chromenes derivative with excellent product separation under the present method. Thus, this green protocol provides an easy method of pure product synthesis without using further chromatographic techniques.

Biological activity: *in vitro* Antimicrobial activity [52] was carried out by disc diffusion method and selected 2-amino-4*H*-chromenes (**3b**, **3d**, **3g**) showed the activity at higher concentrations and are comparable with ciprofloxacin and fluconazole as standards (Table-2). Surprisingly, fluro substituted benzaldehyde derivative of chromene (**3d**) showed a broad antimicrobial activity as compared to compounds **3b** and **3g**. The chromene derivative **3b** which is also halogen substituted but not showed activity like flurine another chromene **3g**, which is more hydrophobic not showed activity from these antimicrobial assay revealed that highly electronegative substituted is showed activity compare to less electronegative and hydrophobic aldehyde (Table-2).

Conclusion

In the present work, agro-waste stuff ash extracted from banana peels (WEB) as a green catalyst for the synthesis of 2amino-4*H*-chromene derivatives is reported. For the synthesis employed substituted benzaldehyde, malononitrile and resorcinol or α/β -naphthol forms under microwave irradiation and grindstone methods. The method offers simple, clean, efficient and economic technique for the synthesis of bioactive scaffold chromenes through green chemistry. Further selected chromenes derivative tested *in vitro* antibacterial and antimicrobial assay in disc-diffusion method and found compounds **3b**, **3d**, **3g**) are active at higher concentration and comparable with standard.

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