Indian Journal of Chemistry
Vol. 59B, June 2020, pp. 850-855

An effortless microwave synthesis of N-(aryl) substituted benzamides under solvent free conditions

Sri Latha Rao[a], Veerabhadraswamy M*[b] & Bhimashankar B Molkere[b]
[a] Department of Chemistry, Nitte Meenakshi Institute of Technology, Bengaluru 560 064, India
[b] Green Chemistry Center, P E S University, Bengaluru 560 085, India
E-mail: vbs@pes.edu

Received 13 February 2019; accepted (revised) 10 February 2020

Benzamides and substituted benzamides are versatile compounds used in a range of pharmacological and biological active intermediate chemicals for conversion to fine chemicals. The current syntheses protocols are chemical and energy intensive. Herein is present an effortless solvent free microwave assisted synthesis of twenty N-(aryl)-substituted benzamides having good to excellent yields with short reaction time, high selectivity and occurring under milder conditions with high atom efficiency. The structures of the prepared compounds have been confirmed using melting point, FT-IR, ^{1}H and ^{13}C NMR spectral data which are in good agreement with the previously reported literature. Unlike conventional methods, the present novel process protocol offers cleaner, greener technologies in which effluent streams are minimized.

Keywords: Anilines, acid chlorides, microwave irradiation, benzamides, characterization

The overview of microwave heating has greatly impacted many aspects of chemical synthesis. There are some excellent reviews and reports on the broad for use of microwave irradiation in organic synthesis[1]. Which constitute an emerging green technology for industrially important organic synthesis more eco-friendly than conventional reactions[2] and more efficient source of heating than conventional systems[3,4], use of microwave heating was intensely cut down reaction time, increase product purity, yields, allow precise control of reaction conditions, simple to handle, reduce pollution, comparatively cheaper to operate, especially vital in pharmaceutical industry, all of which make it suited to light the increased demands of high quantitative and qualitative chemistry. Microwave experiments were performed in such a way that minimum bio-products, have no effect on the environment and their disposal is convenient[5].

Several structurally diverse amides were synthesized by reaction of the corresponding amines with benzoyl chloride under microwave irradiation. The proposed procedure ensures short reaction time, high yields, excellent selectivity and considerably broadens the series of amines as compared to the microwave-assisted synthesis of amides directly from carboxylic acids. It can also be used for selective protection of various amines, including aromatic, aliphatic, and heterocyclic.

Yanqiu Lia[6] describes Microwave-Assisted Synthesis of Amides from Various Amines and Benzoyl Chloride. Albericio *et al.*[7] explains the synthesis of benzamides under solvent free condition; synthesis of amides directly from carboxylic acid by Singh GS[8]; Sheehan J C[9] selective protection of various amines. In the last decade, microwaves (MWs) have been used to simplify and improve reaction conditions for many classical organic reactions. Reactions performed under MW conditions proceed faster, more cleanly, and in much better yields than do similar reactions under conventional conditions[10-15]. Kohli *et al.*[16] explains the infrared studies of some amides.

We extant here a swift and proficient method for the synthesis of aryl substituted benzamides in solvent-free conditions under microwave irradiation. The procedure includes mixture of reactants, aniline and acid chloride in a crucible, followed by exposure to microwave irradiation furnished the corresponding aryl substituted benzamide III a to III t. The structure of compounds III was confirmed by their spectroscopic (FTIR, ^{1}H NMR, and ^{13}C NMR) and analytical data (Table I).

Result and Discussion

The synthesized compounds were characterized by using FTIR, ^{1}H NMR and ^{13}C NMR spectral data, the infrared absorption frequencies of N-(Aryl) substituted benzamides (**IIIa-IIIt**, Figure 1) are in good agreement and conformity with the previously reported literature. The powdered potassium bromide was used for background correction for IR spectrum. The C=O stretching vibrations appear as very strong absorptions in the ranges 1654.8 cm^{-1} and 1649.0 cm^{-1}. The N-H stretching vibrations appear as strong vibrations in the ranges 3246.3-3384.8 cm^{-1}. Similarly, the C-N stretching vibrations absorb in the ranges, 1315.4-1294.1 cm^{-1}. The other frequencies are assigned to various vibrations and have good correlation with reported values.

The observed ^{1}H and ^{13}C chemical shift values of all benzamides (III a- IIIt) were assigned, self-consistency among the observed and calculated values. The incremental shifts of the aromatic protons due to C_6H_5CONH- in C_6H_5CONH (C_6H_5) were calculated by comparing the proton chemical shifts of this compound with the benzene proton value of 7.27 ppm. The calculated incremental shifts due to C_6H_5CONH- are 0.49, 0.03 and -0.20 ppm for H-2, 6: H-3, 5 and H - 4, respectively. Similarly, the incremental shifts of the aromatic protons due to C_6H_5CO- in $C_6H_5CONH(C_6H_5)$ were calculated by comparing the proton chemical shifts of this compound with the aniline proton values of H-2,6 = 6.48 ppm; H-3,5 = 7.05 ppm; H-4 = 6.67.

The computed incremental shifts due to C_6H_5CONH- are 1.28, 0.25 and 0.40 ppm for H-2,6; H-3,5 and H-4 respectively, the chemical shifts of all the aromatic protons in all benzamides were calculated in three different ways by adding either the substituent contributions to the corresponding ^{1}H chemical shifts of N-(phenyl)-benzamide, $C_6H_5CONH(C_6H_5)$ or by adding the incremental shifts due to C_6H_5CONH- and C_6H_5CO- groups to the ^{1}H chemical shifts of the corresponding substituted benzenes and the substituent effects is quite good even for ^{13}C chemical shifts.

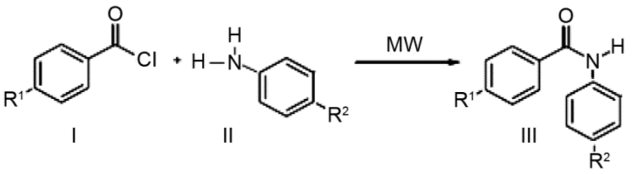
Table I — Characterization of compounds IIIa to IIIt

Compd	Reaction time (min)	m.p. °C	Yield (%)	Mol. formula
IIIa	3.5	124-126	92	$C_{14}H_{12}N_2O_4$
IIIb	3.5	108-110	93	$C_{14}H_{12}BrNO_2$
IIIc	3.5	121-123	90	$C_{14}H_{11}$ ClN_2O_4
IIId	3.5	98-100	93	$C_{15}H_{15}NO3$
IIIe	3.5	162-164	92	$C_{14}H_{12}N_2O_3$
IIIf	3.5	190-192	93	$C_{14}H_{12}BrNO$
IIIg	3.5	157-159	95	$C_{14}H_{11}ClN_2O_3$
IIIh	3.5	86-89	96	$C_{15}H_{15}NO_2$
IIIi	3.5	192-194	93	$C_{13}H_9ClN_2O_3$
IIIj	3.5	203	93	$C_{13}H_9ClBrNO$
IIIk	3.5	134-136	93	$C_{13}H_8Cl_2N_2O_3$
IIIl	3.5	117	93	$C_{14}H_{12}ClNO_2$
IIIm	3.5	131	82	$C_{13}H_9BrN_2O_3$
IIIn	3.5	172-174	80	$C_{13}H_9Br_2NO$
IIIo	3.5	199-201	80	$C_{13}H_8ClBrN_2O_3$
IIIp	3.5	124	77	$C_{14}H_{12}BrNO_2$
IIIq	3.5	140-142	75	$C_{13}H_9FN_2O_3$
IIIr	3.5	206	72	$C_{13}H_9FBrNO$
IIIs	3.5	180-182	70	$C_{13}H_8ClFN_2O_3$
IIIt	3.5	186	78	$C_{14}H_{12}FNO_2$

The overall comparison of all calculated and observed chemical shift values of both the aromatic protons and carbons revealed that the different procedures of calculation lead to almost the same values in most cases and in reasonable agreement with the experimental chemical shifts, indicating that the validity of the principle of additivity of the substituent effects is quite good in these compounds.

Experimental Section

All the reagents employed in the preparations and purification was of commercial anilines (Sigma Aldrich Research Laboratories, India) and purified by either double distillation or zone refining of analytical grade. The reaction course has been carefully monitored and checked by precoated TLC plates. On completion of reaction the crude product (a powder) N-aryl- substituted benzamides were washed thoroughly with water and recrystallized using 95% alcohol and dried under vacuum. The infrared spectra (IR) of aryl-substituted benzamides (IIIa to IIIt) were recorded in KBr pellets (13mm) on a SHIMADZU-8700 FT-IR spectrometer, with a resolution set to 4 cm^{-1} and scan range was from 400–4000 cm^{-1} respectively, the comparison was done with the available literature data. The ^{1}H NMR spectrum on BRUKER Ac 300F, 300MHz FT-NMR spectrometer



	R^1	R^2		R^1	R^2
a	4-OCH_3	4-NO_2	k	4-Cl	2-Cl,4-NO_2
b	4-OCH_3	4-Br	l	4-Cl	OCH_3
c	4-OCH_3	2-Cl,4-NO_2	m	4-Br	4-NO_2
d	4-OCH_3	OCH_3	n	4-Br	4-Br
e	4-CH_3	4-NO_2	o	4-Br	2-Cl,4-NO_2
f	4-CH_3	4-Br	p	4-Br	OCH_3
g	4-CH_3	2-Cl,4-NO_2	q	4-F	4-NO_2
h	4-CH_3	OCH_3	r	4-F	4-Br
i	4-Cl	4-NO_2	s	4-F	2-Cl,4-NO_2
j	4-Cl	4-Br	t	4-F	OCH_3

Figure 1 — Synthetic route for the compounds **IIIa-IIIt**

using $CDCl_3$ and DMSO with tetramethyl silane (Me_4Si) as internal standard and ^{13}C NMR spectrum using $CDCl_3$ and DMSO with tetramethyl silane as the external reference standard.

General procedure for the synthesis of N-(aryl)-substituted benzamides [IIIa to IIIt]

The reactants aniline, 250mg (2 mmol) and acid chloride, 250 mg (2 mmol) were thoroughly mixed in a crucible and exposure to microwave irradiation under solvent-free conditions using modified domestic microwave at 495 W for 3.5 min (Table I). The reaction course was carefully monitored by thin layer chromatogram (TLC) to regulate the reactant ratio, irradiation time, and microwave power level to achieve the maximum yield. The crude product (a powder), N-aryl- substituted benzamides thus obtained was washed with water and purified by recrystallization using 95% alcohol and dried under vacuum (Table I).

Spectral data

IIIa: IR (KBr): 3410.2 m(N-H Symmetric stretch), 3040.2 w (C-H Aromatic symmetric stretch), 2920.0 w, 2830.0 w (C-H Alkane stretch), 1606.8 s (C=O Stretch), 1598.9 w, 1525.6 m (C=C Aromatic in plane stretch), 1436.9 w (N-H in plane bend), 1317.3 w (C-N Stretch), 1028.0 w (C-H Aromatic in plane bend), 827.4 w, 667.3 w (C-H Aromatic out of plane bend), 750.3 m (N-H out of plane bend) and 503.4 cm^{-1} w (C=C Aromatic out of plane stretch); ^1H NMR ($CDCl_3$+ DMSO-d_6): δ 3.75 (s, 3H,CH_3), 6.93-7.85 (m, 4H, Ar-H), 7.89-8.17 (m, 4H, Ar-H), 8.05(s, 1H, NH); ^{13}C NMR ($CDCl_3$ + DMSO-d_6): δ 55.9, 114.4, 121.3, 121.3, 122.5, 122.5, 126.5, 128.5, 142.0, 144.0, 164.1, 164.9.

IIIb: IR (KBr): 3410.5 m (N-H Symmetric stretch), 3062.7 w (C-H Aromatic symmetric stretch), 2918.1 w, 2862.2 w (C-H Alkane stretch), 1600.9 s (C=O Stretch), 1587.3 w, 1523.7 m (C=C Aromatic in plane stretch), 1433.0m (N-H in plane bend), 1303.7 m (C-N Stretch), 1112.9 w (C-H Aromatic in plane bend), 1043.4 w (C-X Stretch), 839.0 w, 650.0 w (C-H Aromatic out of plane bend), 750.3 m (N-H out of plane bend) and 443.6 cm^{-1} w (C=C Aromatic out of plane stretch); ^1H NMR ($CDCl_3$ + DMSO-d_6): δ 3.70 (s, 3H,CH_3), 6.45-6.95 (m, 4H, Ar-H), 7.42-7.85 (m, 4H, Ar-H), 7.89 (s, 1H, NH); ^{13}C NMR ($CDCl_3$ + DMSO-d_6): δ 54.6, 117.6, 121.0, 121.3, 121.5, 123.5, 124.5, 129.8, 141.5, 142.6, 162.5, 165.9.

IIIc: IR (KBr): 3367.7 m (N-H Symmetric stretch), 3055.5 w (C-H Aromatic symmetric stretch), 2920.0 w, (C-H Alkane stretch), 1664.0 s (C=O Stretch), 1587.3m, 1521.7 m (C=C Aromatic in plane stretch), 1413.7m (N-H in plane bend), 1307.6 m (C-N Stretch), 1105.5 w (C-H Aromatic in plane bend), 1076.2 w (C-X Stretch), 850.5 w, 677.0 w (C-H Aromatic out of plane bend), 755.3 m (N-H out of plane bend) and 439.7 cm^{-1} w (C=C Aromatic out of plane stretch); ^1H NMR ($CDCl_3$ + DMSO-d_6): δ 3.65 (s, 3H,CH_3), 6.98-7.89 (m, 4H, Ar-H), 7.85-8.20, 8.06 (bs, 3H, Ar-H), 8.25 (s, 1H, NH); ^{13}C NMR ($CDCl_3$ + DMSO-d_6): δ 55.8, 114.6, 119.5, 123.9, 124.2, 126.1, 128.5, 131.4, 141.0, 145.4, 164.3, 164.7.

IIId: IR (KBr): 3334.3 m (N-H Symmetric stretch), 3060.2 w (C-H Aromatic symmetric stretch), 2919.2 w, (C-H Alkane stretch), 1644.8 s (C=O Stretch), 1586.1w, 1517.9s (C=C Aromatic in plane stretch), 1396.4m (N-H in plane bend), 1309.6 w (C-N Stretch), 1016.4 w (C-H Aromatic in plane bend), 1091.6 w (C-X Stretch), 825.5 m, 640.3m (C-H Aromatic out of plane bend), 748.3 m (N-H out of plane bend) and 507.2 cm^{-1} w (C=C Aromatic out of plane stretch); ^1H NMR ($CDCl_3$ + DMSO-d_6): δ 3.78 (s, 6H,CH_3), 6.75-7.82 (m, 4H, Ar-H), 7.53-7.95 (m, 4H, Ar-H), 8.85 (s, 1H, NH); ^{13}C NMR ($CDCl_3$ + DMSO-d_6): δ 55.6, 55.9, 114.4, 114.5, 114.5, 122.6, 122.6, 126.5, 128.2, 128.5, 156.3, 164.1, 165.8.

IIIe: IR (KBr): 3332.8 m (N-H Symmetric stretch), 3069.2 w (C-H Aromatic symmetric stretch), 2912.1 w, (C-H Alkane stretch), 1653.8 s (C=O Stretch), 1616.2 w, 1523.7m (C=C Aromatic in plane stretch), 1404.1m (N-H in plane bend), 1317.3 w (C-N Stretch), 1022.2 w (C-H Aromatic in plane bend), 1213.1m (C-X Stretch), 831.3w, 640.3w (C-H Aromatic out of plane bend), 748.3w (N-H out of plane bend) and 452.1 cm^{-1} w (C=C Aromatic out of plane stretch); ^1H NMR ($CDCl_3$ + DMSO-d_6): δ 2.45 (s, 3H,CH_3), 7.25-7.95 (m, 4H, Ar-H), 7.99-8.03 (m, 4H, Ar-H), 8.23(s, 1H, NH); ^{13}C NMR ($CDCl_3$ + DMSO-d_6): δ 25.9, 121.5, 121.5, 122.5, 128.5, 128.5, 129.6, 132.9, 142.6, 146.6, 165.9, 166.9.

IIIf: IR (KBr): 3346.3 m (N-H Symmetric stretch), 3053.6 w (C-H Aromatic symmetric stretch), 2925.8w, 2885.5w (C-H Alkane stretch), 1654.8 s (C=O Stretch), 1595.0 w, 1521.7m (C=C Aromatic in plane stretch), 1392.5w (N-H in plane bend), 1311.5w (C-N Stretch), 1010.6 w (C-H Aromatic in plane bend), 1087.7w (C-X Stretch), 821.6m, 655.3w (C-H Aromatic out of plane bend), 750.3m (N-H out of plane bend) and 499.2 cm^{-1} w (C=C Aromatic out of plane stretch); ^1H NMR ($CDCl_3$ + DMSO-d_6): δ 2.50

(s, 3H,CH_3), 7.25-7.55 (m, 4H, Ar-H), 7.88-7.99 (m, 4H, Ar-H), 8.08(s, 1H, NH); ^{13}C NMR ($CDCl_3$ + DMSO-d_6): δ 24.6, 119.5, 122.9, 122.9, 124.3, 124.3, 128.9, 130.4, 132.5, 135.4, 142.4, 165.2.

IIIg: IR (KBr): 3431.0 m (N-H Symmetric stretch), 3093.2 w (C-H Aromatic symmetric stretch), 2925.8w, 2856.4w (C-H Alkane stretch), 1650.2 s (C=O Stretch), 1504.4m (C=C Aromatic in plane stretch), 1493.3m (N-H in plane bend), 1315.4m (C-N Stretch), 1028.0 w (C-H Aromatic in plane bend), 837.0w (C-H Aromatic out of plane bend), 750.3w (N-H out of plane bend) and 501.5 cm^{-1} w (C=C Aromatic out of plane stretch)); ^{1}H NMR ($CDCl_3$ + DMSO-d_6): δ 2.44 (s, 3H,CH_3), 7.19-7.20 (m, 4H, Ar-H), 7.89-8.02 (bs, 3H, Ar-H), 8.23 (s, 1H, NH); ^{13}C NMR ($CDCl_3$ + DMSO-d_6): δ 21.6, 120.0, 121.6, 124.4, 128.5, 128.8, 130.3, 132.2, 141.1, 142.5, 145.5, 165.4.

IIIh: IR (KBr): 3301.3 m (N-H Symmetric stretch), 3022.4 w (C-H Aromatic symmetric stretch), 2930.2w, 2860.2w (C-H Alkane stretch), 1657.0 s (C=O Stretch), 1598.9w, 1546.8s (C=C Aromatic in plane stretch), 1434.9m (N-H in plane bend), 1309.6w (C-N Stretch), 1028.0 w (C-H Aromatic in plane bend), 835.1w, 682.8m (C-H Aromatic out of plane bend), 781.1m (N-H out of plane bend) and 480.2 cm^{-1} w (C=C Aromatic out of plane stretch)); ^{1}H NMR ($CDCl_3$ + DMSO-d_6): δ 2.35, 3.88 (s, 6H, CH_3), 6.88-7.32 (m, 4H, Ar-H), 7.55-7.95 (m, 4H, Ar-H), 8.56 (s, 1H, NH); ^{13}C NMR ($CDCl_3$ + DMSO-d_6): δ 24.8, 56.9, 115.5, 124.6, 124.6, 128.9, 128.9, 128.9, 129.6, 131.6, 142.2, 156.6, 165.5.

IIIi: IR (KBr): 3283.2 m (N-H Symmetric stretch), 3030.2 w (C-H Aromatic symmetric stretch), 2912.0w, 2833.0w (C-H Alkane stretch), 1655.8 s (C=O Stretch), 1538.9w, 1515.6m (C=C Aromatic in plane stretch), 1433.9w (N-H in plane bend), 1313.3w (C-N Stretch), 1028.0 w (C-H Aromatic in plane bend), 1091.6w (C-X Stretch), 826.4w, 657.3w (C-H Aromatic out of plane bend), 740.3m (N-H out of plane bend) and 503.4 cm^{-1} w (C=C Aromatic out of plane stretch); ^{1}H NMR ($CDCl_3$ + DMSO-d_6): δ 7.5 (m, 4H, Ar-H), 7.89-8.03 (m, 4H, Ar-H), 8.23(s, 1H, NH); ^{13}C NMR ($CDCl_3$ + DMSO-d_6): δ 119.9, 121.3, 121.3, 122.2, 122.2, 128.8, 132.2, 138.9, 143.2, 145.5, 165.5.

IIIj: IR (KBr): 3348.5 m (N-H Symmetric stretch), 3043.7 w (C-H Aromatic symmetric stretch), 2928.1 w, 2842.2w (C-H Alkane stretch), 1652.9 s (C=O Stretch), 1567.3w, 1533.7m (C=C Aromatic in plane stretch), 1434.0m (N-H in plane bend), 1303.7 m (C-N Stretch), 1112.9 w (C-H Aromatic in plane bend), 1043.4w (C-X Stretch), 838.0w, 650.0w (C-H Aromatic out of plane bend), 770.3m (N-H out of plane bend) and 443.6 cm^{-1} w (C=C Aromatic out of plane stretch); ^{1}H NMR ($CDCl_3$ + DMSO-d_6): δ 7.40 (m, 4H, Ar-H), 7.54-7.95 (m, 4H, Ar-H), 8.54(s, 1H, NH); ^{13}C NMR ($CDCl_3$ + DMSO-d_6): δ 119.9, 124.5, 124.6, 124.6, 128.7, 128.7, 129.9, 132.2, 135.5, 138.5, 165.6.

IIIk: IR (KBr): 3342.7 m (N-H Symmetric stretch), 2912.0 w (C-H Alkane stretch), 1652.0 s (C=O Stretch), 1577.3w, 1522.7m (C=C Aromatic in plane stretch), 1412.7m (N-H in plane bend), 1305.6 m (C-N Stretch), 1105.5 w (C-H Aromatic in plane bend), 1076.2w (C-X Stretch), 851.5w, 627.0w (C-H Aromatic out of plane bend), 755.3m (N-H out of plane bend) and 439.7 cm^{-1} w (C=C Aromatic out of plane stretch); ^{1}H NMR ($CDCl_3$ + DMSO-d_6): δ 7.45 (m, 4H, Ar-H), 7.89-8.56 (bs, 3H, Ar-H), 8.52(s, 1H, NH); ^{13}C NMR ($CDCl_3$ + DMSO-d_6): δ 119.6, 122.5, 124.5, 127.9, 128.9, 131.2, 132.2, 138.8, 141.3, 145.0, 165.0.

IIIl: IR (KBr): 3280.3 m (N-H Symmetric stretch), 3066.2 w (C-H Aromatic symmetric stretch), 2923.2w (C-H Alkane stretch), 1641.8 s (C=O Stretch), 1586.1w, 1515.9s (C=C Aromatic in plane stretch), 1398.4m (N-H in plane bend), 1309.6 w (C-N Stretch), 1016.4 w (C-H Aromatic in plane bend), 825.5m, 620.3m (C-H Aromatic out of plane bend), 758.3m (N-H out of plane bend) and 507.2 cm^{-1} w (C=C Aromatic out of plane stretch); ^{1}H NMR ($CDCl_3$ + DMSO-d_6): δ 3.75 (s, 3H,CH_3), 6.75-7.55 (m, 4H, Ar-H), 7.45-7.95 (m, 4H, Ar-H), 8.03(s, 1H, NH); ^{13}C NMR ($CDCl_3$ + DMSO-d_6): δ 55.63, 115.6, 124.5, 124.5, 128.9, 129.8, 129.9, 129.9, 136.9, 138.9, 158.8, 164.4.

IIIm: IR (KBr): 3298.8 m (N-H Symmetric stretch), 3069.2 w (C-H Aromatic symmetric stretch), 2912.1w (C-H Alkane stretch), 1651.8 s (C=O Stretch), 1516.2w, 1521.7m (C=C Aromatic in plane stretch), 1402.1m (N-H in plane bend), 1316.3w (C-N Stretch), 1022.2 w (C-H Aromatic in plane bend), 1213.1m (C-X Stretch), 831.3w, 650.3w (C-H Aromatic out of plane bend), 728.3w (N-H out of plane bend) and 452.1 cm^{-1} w (C=C Aromatic out of plane stretch); ^{1}H NMR ($CDCl_3$ + DMSO-d_6): δ 7.29-8.02 (m, 4H, Ar-H), 7.85-7.89 (m, 4H, Ar-H), 8.29(s, 1H, NH); ^{13}C NMR ($CDCl_3$ + DMSO-d_6): δ 115.5, 121.3, 121.3, 122.2, 122.2, 129.6, 130.3, 142.6, 144.6, 165.5, 168.8.

IIIn: IR (KBr): 3346.3 m (N-H Symmetric stretch), 2933.8w,2865.5w (C-H Alkane stretch), 1654.8s (C=O Stretch), 1585.0w, 1522.7m (C=C Aromatic in plane stretch), 1398.5w (N-H in plane bend), 1313.5w (C-N Stretch), 1010.6 w (C-H Aromatic in plane bend), 1087.7w (C-X Stretch), 821.6m, 635.3w (C-H Aromatic out of plane bend), 740.3m (N-H out of plane bend) and 499.2 cm^{-1} w (C=C Aromatic out of plane stretch); ^{1}H NMR ($CDCl_3$ + DMSO-d_6): δ 7.14- 7.55 (m, 4H, Ar-H), 7.45-7.98 (m, 4H, Ar-H), 8.56(s, 1H, NH); ^{13}C NMR ($CDCl_3$ + DMSO-d_6): δ 116.6, 118.8, 118.8, 123.6, 123.6, 129.8, 130.3, 132.5, 135.5, 164.5, 167.8.

IIIo: IR (KBr): (cm-1): 3268.0 m (N-H Symmetric stretch), 3073.2w (C-H Aromatic symmetric stretch), 2925.8w, 2856.4w (C-H Alkane stretch), 1647.2s (C=O Stretch), 1544.4m (C=C Aromatic in plane stretch), 1408.3m (N-H in plane bend), 1314.4m (C-N Stretch), 1028.0 w (C-H Aromatic in plane bend), 1078.7w (C-X Stretch), 857.0w (C-H Aromatic out of plane bend), 755.3 cm^{-1} w (N-H out of plane bend) and 501.5w (C=C Aromatic out of plane stretch); 1H NMR (CDCl3 + DMSO-d_6): δ 7.14-7.35 (m, 4H, Ar-H), 7.35-7.95 (bs, 3H, Ar-H), 8.18(s, 1H, NH); 13C NMR (CDCl3 + DMSO-d_6): δ 115.5, 115.6, 122.2, 123.2, 123.5, 130.5, 132.2, 142.2, 148.5, 168.5, 201.3.

IIIp: IR (KBr): 3286.3 m (N-H Symmetric stretch), 3022.4w (C-H Aromatic symmetric stretch), 2923.2w, 2862.2w (C-H Alkane stretch), 1651.0s (C=O Stretch), 1592.9w, 1542.8s (C=C Aromatic in plane stretch), 1404.9m (N-H in plane bend), 1308.6w (C-N Stretch), 1028.0 w (C-H Aromatic in plane bend), 855.1w, 632.8m (C-H Aromatic out of plane bend), 771.1m (N-H out of plane bend) and 480.2 cm^{-1} w (C=C Aromatic out of plane stretch); ^{1}H NMR ($CDCl_3$ + DMSO-d_6): δ 3.77 (s, 3H,CH_3), 6.28-7.55 (m, 4H, Ar-H), 7.25-7.95 (m, 4H, Ar-H), 8.55(s, 1H, NH); ^{13}C NMR ($CDCl_3$ + DMSO-d_6): δ 56.5, 114.5, 114.5, 115.8, 122.5, 122.5, 129.8, 130.5, 131.2, 155.5, 164.6, 165.9.

IIIq: IR (KBr): 3359.8 m (N-H Symmetric stretch), 3030.2 w (C-H Aromatic symmetric stretch), 2912.0w (C-H Alkane stretch), 1654.8 s (C=O Stretch), 1577.3m (C=C Aromatic in plane stretch), 1398.5w (N-H in plane bend), 1305.6m (C-N Stretch), 1028.0 w (C-H Aromatic in plane bend), 1076.2w (C-X Stretch), 841.5w (C-H Aromatic out of plane bend), 740.3m (N-H out of plane bend) and 444.6 cm^{-1} w (C=C Aromatic out of plane stretch)); ^{1}H NMR ($CDCl_3$ + DMSO-d_6): δ 7.68-7.95 (m, 4H, Ar-H), 7.89-8.05 (m, 4H, Ar-H), 8.20(s, 1H, NH); ^{13}C NMR ($CDCl_3$ + DMSO-d_6): δ 121.3, 122.3, 122.3, 127.8, 127.8, 130.3, 132.2, 133.6, 142.3, 143.6, 163.0.

IIIr: IR (KBr): 3290.5 m (N-H Symmetric stretch), 3022.4 w (C-H Aromatic symmetric stretch), 2923.2w (C-H Alkane stretch), 1645.8 s (C=O Stretch), 1515.9s (C=C Aromatic in plane stretch), 1408.3m (N-H in plane bend), 1309.6m (C-N Stretch), 1112.9 w (C-H Aromatic in plane bend), 1091.6w (C-X Stretch), 853.5w (C-H Aromatic out of plane bend), 770.3m (N-H out of plane bend) and 438.7 cm^{-1} w (C=C Aromatic out of plane stretch); ^{1}H NMR ($CDCl_3$ + DMSO-d_6): δ 7.45-7.68 (m, 4H, Ar-H), 7.57-7.89 (m, 4H, Ar-H), 8.03(s, 1H, NH); ^{13}C NMR ($CDCl_3$ + DMSO-d_6): δ 118.8, 119.5, 119.5, 124.6, 124.6, 127.8, 130.5, 132.5, 133.3, 135.6, 164.6.

IIIs: IR (KBr): 3348.2 m (N-H Symmetric stretch), 2912.1w (C-H Alkane stretch), 1652.2s (C=O Stretch), 1521.7m (C=C Aromatic in plane stretch), 1398.4m (N-H in plane bend), 1316.3w (C-N Stretch), 1105.5 w (C-H Aromatic in plane bend), 1213.1m (C-X Stretch), 850.5w (C-H Aromatic out of plane bend), 755.3m (N-H out of plane bend) and 501.2 cm^{-1} w (C=C Aromatic out of plane stretch); ^{1}H NMR ($CDCl_3$ + DMSO-d_6): δ 7.65-7.88 (m, 4H, Ar-H), 7.54-8.15 (bs, 3H, Ar-H), 8.26(s, 1H, NH); ^{13}C NMR ($CDCl_3$ + DMSO-d_6): δ 119.8, 119.8, 124.8, 129.5, 130.5, 132.5, 134.5, 135.6, 141.5, 145.5, 164.5.

IIIt: IR (KBr): 3122.2 m (N-H Symmetric stretch), 3023.4w (C-H Aromatic symmetric stretch), 2933.8w, 2865.5w (C-H Alkane stretch), 1645.0s (C=O Stretch), 1522.7m (C=C Aromatic in plane stretch), 1412.7m (N-H in plane bend), 1313.5w (C-N Stretch), 1016.4 w (C-H Aromatic in plane bend), 849.5w (C-H Aromatic out of plane bend), 758.3m (N-H out of plane bend) and 482.1 cm^{-1} w (C=C Aromatic out of plane stretch); ^{1}H NMR ($CDCl_3$ + DMSO-d_6): δ 3.78 (s, 3H,CH_3), 6.78-7.65 (m, 4H, Ar-H), 7.52-7.84 (m, 4H, Ar-H), 8.05(s, 1H, NH); ^{13}C NMR ($CDCl_3$ + DMSO-d_6): δ 56.8, 115.8, 120.5, 120.5, 127.8, 127.8, 129.8, 130.9, 132.5, 134.5, 157.8, 164.8.

Conclusion

In summary, a novel series of substituted benzamides were easy synthesised using microwave and the characterisation of these synthesised compounds [IIIa to IIIt] showed good in agreement and conformity. We have utilized non-conventional methodologies for the synthesis of N-(Aryl) substituted benzamides under solvent free conditions and this method is attractive due to its simplicity, ease of workability, enhanced yields, reduced reaction time and practical applicability. Hence, this study provides an essential casing effort, which can be explored for the development of pharmacological and biological active intermediate chemicals for organic conversion to fine chemicals.

Acknowledgement

Authors greatly appreciate the support of the Visvesvaraya Technological University (VTU), Belagavi, Karnataka, and All India Council for Technical Education (AICTE), India and VGST-KFIST (L1) for providing funds for research activity.

References

1 Das S K, *Synlett*, 915 (2004); (b) Lidstrom P, Tierney J, Wathey B & Westman J, *Tetrahedron*, 57 (2001) 9225; (c) Mavandadi F & Lidstrom P, *Curr Top Med Chem*, 4 (2004) 773; (d) Kappe C O, *Angew Chem Int Ed*, 43 (2004) 6250; (e) Yin W, Ma Y, Xu J X & Zhao Y F, *J Org Chem*, 71 (2006) 4312; (f) Cui S L, Lin X F & Wang Y G, *J Org Chem*, 70 (2005) 2866; (g) Mishra J K, Rao J K, Sastry G N & Panda G, *Tetrahedron Lett*, 47 (2006) 3357.

2 Rani A, 'MORE, a Green Technique', *Second International Symposium on Green/Sustainable Chemistry*, http://www.green conference2006.com (2006).

3 Gedye R, Smith F, Westaway K, Ali H, Baldisera L, Laberge L & Rousell J, *Tetrahedron Lett*, 27 (1986) 279.

4 Verma R S, *Pure Appl Chem*, 73 (2001) 193.

5 Kidwai M & Ahluwalia V K, *New Trends in Green Chemistry* (Anamaya Publishers, New Delhi) (2004).

6 Li Y, Wang Y & Wang J, *Russ J Org Chem*, 44(3) (2008) 358.

7 Albericio F, *Curr Opin Chem Biol*, 8 (2004) 211.

8 Singh G S, *Tetrahedron*, 59 (2003) 7631.

9 Sheehan J C & Hess G P *J Am Chem Soc*, 77 (1955) 1067.

10 Loupy A, Petit A, Hamelin J, Texier Boullet F, Jacquault P & Mathé D, *Synthesis*, 1213 (1998).

11 Kappe C O, *Angew Chem Int*, 43 (2004) 6250.

12 Lidstrom P, Tierney J, Wathey B & Westman J, *Tetrahedron*, 57 (2001) 9225.

13 Caddik S, *Tetrahedron*, 51 (1995) 10403.

14 Kuhnert N, *Angew Chem Int Ed*, 41 (2002) 1863.

15 de la Hoz A, Diaz-Ortiz A & Moreno A, *Chem Soc Rev*, 164 (2005).

16 Kidwai M, *Pure Appl Chem*, 73(1) (2001) 147.