



**MICROWAVE-ASSISTED ONE-POT SYNTHESIS OF BENZO[d] THIAZOLE
CONTAINING 1,2,3-TRIAZOLES BY USING ORGANO CATALYTIC REACTION
AND THEIR ANTIBACTERIAL ACTIVITY**

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ABSTRACT:

Microwave irradiation was used for the synthesis of benzo[d]thiazole containing 1,4,5-trisubstituted 1,2,3-triazoles in one pot, three component organocatalytic cycloadditions of Julia reagent, alkyl halide and sodium azide. The molecular structures of the compounds were determined by use of ¹H NMR, IR, Mass and elemental analysis. The antimicrobial study of these derivatives showed that **3i**, **3b** and **3c** registered good antibacterial activity.

KEYWORDS: Microwave irradiation; One-pot synthesis; 1,2,3-triazoles; Antibacterial activity.

INTRODUCTION

The importance of 1,2,3-triazole derivatives in biological systems has attracted great interest due to their broad spectrum of biological activities and are widely used in organic, medicinal, and material science^{I-V}. Sulfonyl-containing 1,2,3-triazole compounds are of considerable importance in medicinal chemistry. For example, Sulfonyl 1,2,3-triazole moiety possessing clinically using drugs includes β -lactam antibiotics, Tazobactam (**Fig.1**)^{VI} and 1,2,3-triazoles coupled diaryl sulfone moieties were identified as novel small molecule scaffolds for potential antibacterial, antifungal, and antioxidant agents by Mohamed F. Mady group^{VII}. On the other hand, the chemistry of benzo[d]thiazole scaffolds has received considerable attention owing to their synthetic and effective biological importance (**Fig.1**)^{VIII-X}.

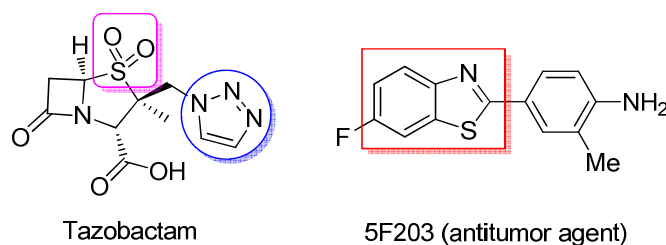
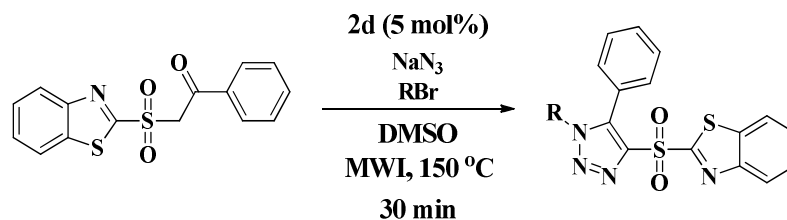


Figure 1: Examples of 1,2,3-triazole and benzo[d]thiazole-based biologically active compounds.

The synthesis of 1,4- and 1,5-disubstituted 1,2,3-triazoles can be achieved by copper-catalyzed [3+2]cycloaddition^{XI, XII} and ruthenium-catalyzed [3+2] cycloaddition reaction^{XIII-XIV} respectively. Recently, an enamine-mediated amino acid or amine catalyzed [3+2] cycloaddition reaction of different carbonyl compounds with aryl azides was reported to furnish 1,4,5-trisubstituted 1,2,3-triazoles in good yields^{XV-XVIII}. Very recently, Changhu Chu *et al.* reported 4,5-disubstituted 1,2,3-(NH)-triazoles by using one-pot, a three-component reaction using modified Julia reagents^{XIX}. Also in 2015, Diego Alves and co-workers reported organocatalytic cycloaddition of azides with β -keto sulfones^{XX}.

Encouraged by the above mentioned successful synthesis of highly functionalized 1,2,3-triazoles via organocatalytic enamine-azide cycloaddition with organic azides with carbonyl compounds and keeping in view of their versatile therapeutic properties, as well as in continuation of our research on 1,2,3- triazoles^{XXI-XXXI}, herein, we report an efficient method for the synthesis of fully substituted 1,2,3-triazole derivatives by using one pot, three component organocatalytic cycloadditions of Julia reagent, alkyl halides with sodium azide under microwave irradiation (**Scheme 1**).



Scheme 1: Design of the microwave assisted enolate-mediated one-pot [3+2] cycloaddition reaction.

MATERIALS AND METHODS

All the reagents and solvents were purchased from Aldrich/Merck and used without further purifications. Thin-layer chromatography (TLC) was performed using Merck silica gel 60 F254 precoated plates (0.25 mm) and Silica gel (100-200 mesh) was used for column chromatography. The progress of the reactions as well as purity of the compounds was monitored by thin layer chromatography with using ethylacetate /hexane (4/6) as eluent. Melting points were determined using a Cintex apparatus and are uncorrected. 400 MHz spectrometer was used to get ¹H-NMR spectra respectively. Coupling constant (J) values are presented in Hertz, spin multiples are given

as s (singlet), d (doublet), t (triplet), and m (multiplet). Mass spectra were recorded by using ESI-MS method.

Synthesis of 2-((1-alkyl-5-phenyl-1H-1,2,3-triazol-4-yl) sulfonyl) benzo[d]thiazole(3a-3i):

To a mixture of 2-(benzo[d]thiazol-2-ylsulfonyl)-1-phenylethanone **1** (1 mmol), alkyl halide (1.3 mmol) and sodium azide (1.3 mmol) in DMSO (15 mL) in a 50-mL glass vial, was added pyrrolidine (5 mol %) and the vial was tightly sealed with an aluminum/Teflon crimp top. The mixture was then irradiated for 30 min at 150 °C. After completion of the reaction, the vial was cooled to room temperature, and the reaction mixture was poured carefully into ice-cold water (15 mL) and the product was extracted with ethyl acetate (2 x 15 mL). The combined organic layer was dried over anhydrous Na₂SO₄. After filtration, the solvent was evaporated under vacuum and the crude product was purified by the column chromatography (silica gel, 40% ethyl acetate in hexane) to afford 1,4,5-trisubstituted 1,2,3-triazoles in good yields.

2-((1-hexyl-5-phenyl-1H-1,2,3-triazol-4-yl)sulfonyl)benzo[d]thiazole(3a): White solid; mp: 57-59 °C; IR (KBr, cm⁻¹) 2964, 1587, 1472, 1411; ¹H NMR (400 MHz, CDCl₃) 8.11 (d, *J*= 7.6 Hz, 1H), 7.91- 7.84 (m, 3H), 7.60-7.51 (m, 3H), 7.48-7.29 (m, 2H), 4.31(t, *J* = 7.7 Hz, 2H, N-CH₂), 2.01-1.82 (m, 2H), 1.42-1.19 (m, 6H), 0.88 (t, *J* = 7.2 Hz, 3H); Anal. calcd. for C₂₁H₂₂N₄O₂S₂: C, 59.13; H, 5.20; N, 13.13. Found: C, 59.24; H, 5.27; N, 13.07; MS (ESI, m/z):427 [M+H]⁺.

2-((1-heptyl-5-phenyl-1H-1,2,3-triazol-4-yl)sulfonyl)benzo[d]thiazole (3b): White solid; mp: 62-64 °C; IR (KBr, cm⁻¹) 2930, 1575, 1447, 1410; ¹H NMR (400 MHz, CDCl₃) 8.09 (d, *J*= 7.6 Hz, 1H), 7.93- 7.81 (m, 3H), 7.62-7.50 (m, 3H), 7.45-7.26 (m, 2H), 4.35 (t, *J* = 7.3 Hz, 2H, N-CH₂), 1.97-1.80 (m, 2H), 1.42-1.21 (m, 8H), 0.86 (t, *J* = 6.8 Hz, 3H); Anal. calcd. for C₂₂H₂₄N₄O₂S₂: C, 59.97; H, 5.49; N, 12.72. Found: C, 60.04; H, 5.53; N, 12.65; MS (ESI, m/z): 441 [M+H]⁺.

2-((1-octyl-5-phenyl-1H-1,2,3-triazol-4-yl)sulfonyl)benzo[d]thiazole (3c): White solid; mp: 79-81 °C; IR (KBr, cm⁻¹) 2961, 1567, 1452, 1421; ¹H NMR (400 MHz, CDCl₃) 8.11 (d, *J*= 7.6 Hz, 1H), 7.89- 7.80 (m, 3H), 7.62-7.53 (m, 3H), 7.47-7.28 (m, 2H), 4.30 (2H, t, *J*= 7.74 Hz, N-CH₂), 1.95-1.78 (m, 2H), 1.40-1.20 (m, 10H), 0.84 (3H, t, *J* = 7.17 Hz); Anal. calcd. for C₂₃H₂₆N₄O₂S₂: C, 60.77; H, 5.76; N, 12.32. Found: C, 60.83; H, 5.81; N, 12.28; MS (ESI, m/z): 455 [M+H]⁺.

2-((1-decyl-5-phenyl-1H-1,2,3-triazol-4-yl)sulfonyl)benzo[d]thiazole (3d): White solid; mp: 84-86 °C; IR (KBr, cm⁻¹) 2981, 1574, 1452, 1418; ¹H NMR (400 MHz, CDCl₃) 8.10 (d, *J*= 7.8 Hz, 1H), 7.92- 7.81 (m, 3H), 7.61-7.53 (m, 3H), 7.49-7.27 (m, 2H), 4.33 (t, *J*= 7.2 Hz, 2H, N-CH₂), 1.97-1.80 (m, 2H), 1.42-1.23 (m, 14H), 0.85 (t, *J* = 6.8 Hz, 3H); Anal. calcd. for C₂₅H₃₀N₄O₂S₂: C, 62.21; H, 6.26; N, 11.61. Found: C, 62.30; H, 6.29; N, 11.57; MS (ESI, m/z): 483 [M+H]⁺.

2-((1-dodecyl-5-phenyl-1H-1,2,3-triazol-4-yl)sulfonyl)benzo[d]thiazole (3e): White solid; mp: 92-94 °C; IR (KBr, cm⁻¹) 2979, 1557, 1470, 1419; ¹H NMR (400 MHz, CDCl₃) 8.08 (d, *J*= 7.7 Hz, 1H), 7.92- 7.81 (m, 3H), 7.62-7.54 (m, 3H), 7.49-7.28 (m, 2H), 4.32 (t, *J* = 7.6 Hz, 2H, N-CH₂), 1.98-1.81 (m, 2H), 1.46-1.19 (m, 18H), 0.87 (t, *J* = 6.8 Hz, 3H); Anal. calcd. for

C₂₇H₃₄N₄O₂S₂: C, 63.50; H, 6.71; N, 10.97. Found: C, 63.62; H, 6.78; N, 10.93; MS (ESI, m/z): 511 [M+H]⁺.

2-((5-phenyl-1-tridecyl-1H-1,2,3-triazol-4-yl)sulfonyl)benzo[d]thiazole (3f): White solid; mp: 96-98 °C; IR (KBr, cm⁻¹) 2944, 1569, 1461, 1418; ¹H NMR (400 MHz, CDCl₃) 8.09 (d, *J*= 7.8 Hz, 1H), 7.93- 7.81 (m, 3H), 7.61-7.52 (m, 3H), 7.50-7.28 (m, 2H), 4.34 (t, *J* = 7.5 Hz, 2H, N-CH₂), 1.97-1.79 (m, 2H), 1.44-1.18 (m, 20H), 0.83 (t, *J* = 6.9 Hz, 3H); Anal. calcd. for C₂₈H₃₆N₄O₂S₂: C, 64.09; H, 6.91; N, 10.68. Found: C, 64.15; H, 6.93; N, 10.61; MS (ESI, m/z): 525 [M+H]⁺.

2-((5-phenyl-1-tetradecyl-1H-1,2,3-triazol-4-yl)sulfonyl)benzo[d]thiazole(3g): White solid; mp: 103-105 °C; IR (KBr, cm⁻¹) 2955,1562, 1468, 1419; ¹H NMR (400 MHz, CDCl₃) 8.11 (d, *J*= 7.6 Hz, 1H), 7.93- 7.81(m, 3H), 7.62-7.55 (m, 3H), 7.47-7.26 (m, 2H), 4.34 (t, *J* = 7.16 Hz, 2H, N-CH₂-),1.97-1.81 (m, 2H), 1.45-1.16 (m, 22H), 0.84 (t, *J* = 7.1 Hz, 3H); Anal. calcd. for C₂₉H₃₈N₄O₂S₂: C, 64.65; H, 7.11; N, 10.40. Found: C, 64.69; H, 7.17; N, 10.35; MS (ESI, m/z): 539 [M+H]⁺.

2-((1-pentadecyl-5-phenyl-1H-1,2,3-triazol-4-yl)sulfonyl)benzo[d]thiazole(3h): Pale yellow solid; mp: 112-114 °C; IR (KBr, cm⁻¹) 2949, 1561, 1454, 1421; ¹H NMR (400 MHz, CDCl₃) 8.08 (d, *J*= 7.3 Hz, 1H), 7.90- 7.83 (m, 3H), 7.63-7.50 (m, 3H), 7.49-7.25 (m, 2H), 4.31 (t, *J* =7.3 Hz, 2H, N-CH₂-), 1.99-1.81 (m, 2H), 1.44-1.15 (m, 24H), 0.84 (3H, t, *J* = 6.79 Hz). Anal. calcd. for C₃₀H₄₀N₄O₂S₂: C, 65.18; H, 7.29; N, 10.14. Found: C, 65.24; H, 7.33; N, 10.08; MS (ESI, m/z): 553 [M+H]⁺.

2-((1-heptadecyl-5-phenyl-1H-1,2,3-triazol-4-yl)sulfonyl)benzo[d]thiazole(3i): Pale yellow solid; mp: 118-120 °C; IR (KBr, cm⁻¹) 2974, 1565, 1467, 1420; ¹H NMR (400 MHz, CDCl₃) 8.09 (d, *J*= 7.8 Hz, 1H), 7.90- 7.81 (m, 3H), 7.63-7.54 (m, 3H), 7.47-7.25 (m, 2H), 4.33 (t, *J* = 7.5 Hz, 2H, N-CH₂-), 1.94-1.78 (m, 2H), 1.46-1.11 (m, 28H), 0.83 (t, *J* = 7.0 Hz, 3H); Anal. calcd. for C₃₂H₄₄N₄O₂S₂: C, 66.17; H, 7.64; N, 9.65. Found: C, 66.24; H, 7.68; N, 9.61; MS (ESI, m/z): 581 [M+H]⁺.

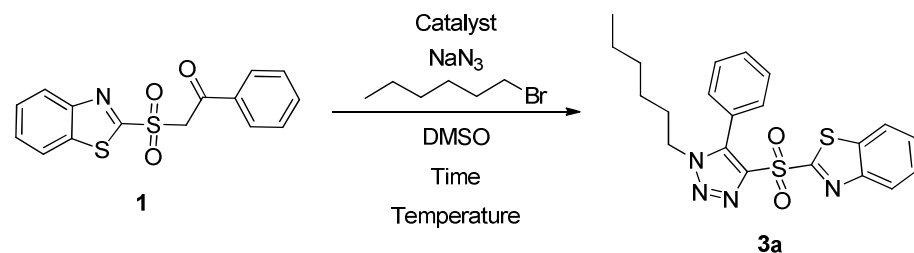
RESULTS AND DISCUSSION

Chemistry

Our interest in developing a new method for the synthesis of highly functionalized 1,2,3-triazoles encouraged us to test the three-component reactions between Julia reagent (2-(benzo[d]thiazol-2-ylsulfonyl)-1-phenylethanone) (**1**) (**Scheme 2**.), alkyl halide and sodium azide with the base under microwave condition. Herein, we would like to describe an efficient method for obtaining 1,4,5-trisubstituted-1,2,3-triazoles (Scheme 1). Our initial investigation was started with one pot [3+2] cycloaddition reaction of 1-bromohexane, sodium azide and Julia reagent (**1**) by following the previously reported literature condition^{XX}. However, the reaction did not proceed and the desired product **3a** was not obtained (Table 1, entry 1). The same reaction was carried out above 100 °C temperature condition by using triethylamine (5 mol %) (**2a**) as catalyst the products were obtained in low yield (Table 1, entry 2). Our investigation of **2b**, **2c**, and **2d** as the catalysts showed that **2d** (5 mol %) was optimal (Table 1, entries 3, 4 and 5). When the same reaction was carried out under microwave irradiation resulted in the generation of the desired product **3a** in an

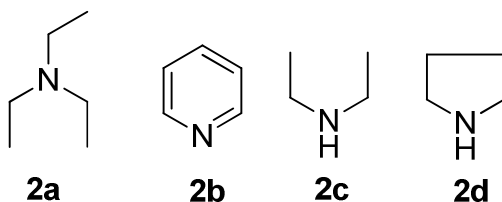
excellent yield (Table 1, entry 6). It was also found that the loadings of catalyst could be increased to 10 mol% almost without affecting the yield of the desired products (Table 1, entry 7). Thus, the combination of 5 mol% of **2d** in the presence of DMSO under microwave irradiation was the optimal reaction conditions.

Table 1. Conditions used for preparation of **3a**^a



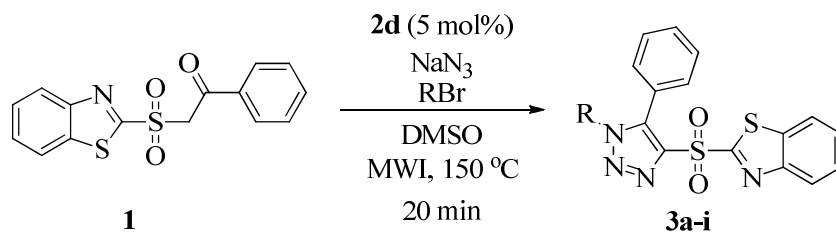
Entry	Catalyst	Temperature (°C)	Time (h)	Yield (%)
1	2d (5 mol %)	Rt	24	NR
2	2a (5 mol %)	>100	24	16
3	2b (5 mol %)	>100	24	32
4	2c (5 mol %)	>100	20	48
5	2d (5 mol %)	>100	18	63
6	2d (5 mol %)	MW/150 °C	30min	81
7	2d (10 mol %)	MW/200 °C	30min	83

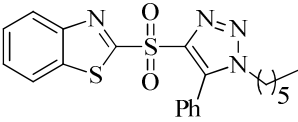
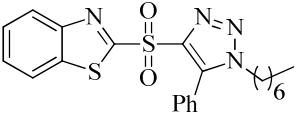
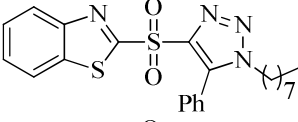
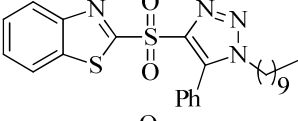
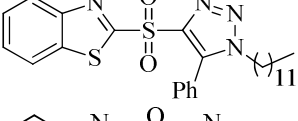
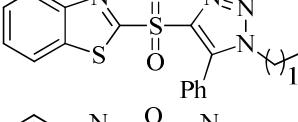
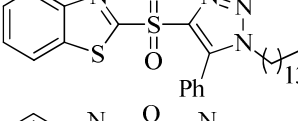
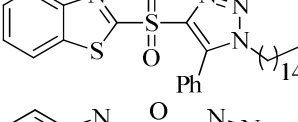
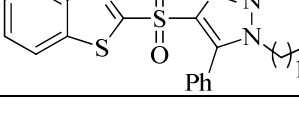
^aReaction conditions: **1** (1.0 equiv), 1-bromohexane (1.3 equiv), NaN₃(1.3 equiv).



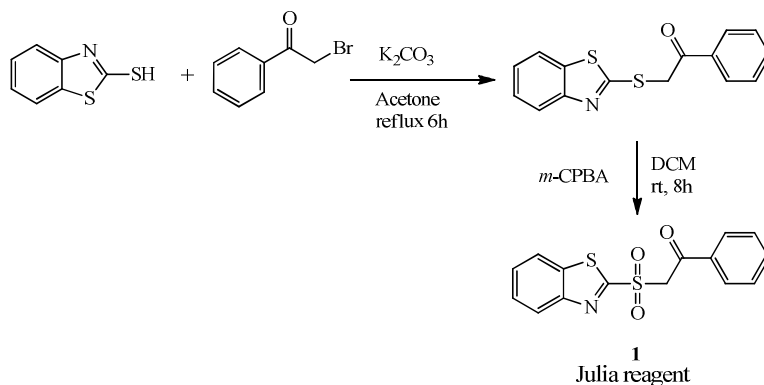
To extend the generality of the reaction, the one pot [3+2] cycloaddition reactions were carried out under the optimized conditions using different alkyl halides. As shown in Table 2, the cycloaddition reaction proceeded well with almost all of these substrates and gave good to excellent yields.

Table 2. Organocatalyzed one-pot synthesis of highly functionalized 1,2,3-triazoles under MWI^a



Compound	Alkyl halide	Product	Yield (%)
3a	C ₆ H ₁₃ Br		81
3b	C ₇ H ₁₅ Br		83
3c	C ₈ H ₁₇ Br		80
3d	C ₁₀ H ₂₁ Br		79
3e	C ₁₂ H ₂₅ Br		80
3f	C ₁₃ H ₂₇ Br		77
3g	C ₁₄ H ₂₉ Br		75
3h	C ₁₅ H ₃₁ Br		72
3i	C ₁₇ H ₃₅ Br		69

^a Isolated yield.



Scheme 2: Synthetic route of compound 1 (Julia reagent)

IN VITRO ANTIBACTERIAL ACTIVITY

The minimum inhibitory concentrations (MIC) of the synthesized compounds (**3a-3i**) were tested against the gram-positive organisms *Bacillus subtilis* and *Staphylococcus aureus* and the gram-negative organisms *Escherichia coli* and *Pseudomonas aeruginosa* using the broth dilution method^{xxxii}. Streptomycin was also screened under identical conditions for comparison. From results obtained (**Table 3**), it is clear that, compound **3i** showed potent activity against *S.aureus* (MIC= 3.12 µg/mL) and the compounds **3b** and **3c** showed good activity against *B.subtilis* (MIC= 6.25 µg/mL). Similarly the compound **3f** exhibited good activity against *E.coli* (MIC= 6.25 µg/mL) and moderate activity against *P.aeruginosa* (MIC= 6.25 µg/mL) when compared with the standard drugs Penicillin and streptomycin.

Table 3. *In vitro* antibacterial activity data of titled compounds (**3a-3i**) as MIC / µg mL⁻¹

Compound	<i>B.subtilis</i>	<i>S.aureus</i>	<i>E.coli</i>	<i>P.aeruginosa</i>
3a	25	>100	>100	>100
3b	6.25	>100	>100	>100
3c	6.25	>100	50	>100
3d	>100	12.5	>100	50
3e	25	50	>100	>100
3f	>100	>100	12.5	12.5
3g	>100	>100	25	>100
3h	>100	>100	>100	>100
3i	>100	3.12	>100	>100
Streptomycin	6.25	6.25	6.25	1.56

CONCLUSION:

In conclusion, we have developed a versatile microwave assisted organocatalytic one-pot three component reaction that generates fully substituted 1,2,3-triazoles. This protocol highlights the metal-free conditions with high regioselectivity, and it provides an easy access to diversely functionalized 1,2,3-triazoles. Newly synthesized triazoles were screened for their anti bacterial activity and **3i**, **3b** and **3c** were found to exhibit good activity compared to standard drug.

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