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Research Article

Cu(II) complexes of 2-methoxy-5-sulfamoylbenzoic acid and 2-aminopyridine derivatives: Synthesis, characterization and antimicrobial activity

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ABSTRACT

Three novel mixed ligand Cu(II) complexes {$[Cu(sba)_2(X)_2].2H_2O$, X = $2a3NO_24mp$ for **1**, 2a5Clp for **2**, $2a5NO_2p$ for **3**} obtained from 2-methoxy-5-sulfamoylbenzoic acid (Hsba) with 2-amino-3-nitro-4-methylpyridine ($2a3NO_24mp$), 2-amino-5-chloropyridine (2a5Clp) and 2-amino-5-nitropyridine ($2a5NO_2p$), respectively, have been prepared. The structures of **1-3** FT-IR, AAS, UV, molar conductivity, magnetic susceptibility, and charge balance as suggested by previous studies have been characterized. According to spectroscopic findings, the structures of **1-3** can be postulated as tetrahedral complexes. Furthermore, all compounds have been screened for their antibacterial and antifungal activities against *Pseudomonas aeruginosa* (ATCC 27853), Enterococcus faecalis (ATCC 29212), *Staphylococcus aureus* (NRRL B-767), *Bacillus subtilis*, *Escherichia coli* (ATCC25922) and *Listeria monocytogenes* (ATCC 7644) bacteria and *Candida albicans* (F89) yeast. The outcomes are contrasted with those of the control substances (Fluconazole as antifungal and Cefepime, Levofloxacin, and Vancomycin as antibacterial agents). The best activity values of the compounds are $Cu(Ac)_2.2H_2O$ for *S. aureus* bacteria, $2a5NO_2p$ for *E. coli* bacteria, **2** for *P. aeruginoa* bacteria, Hsba, $2a3NO_24mp$ and $2a5NO_2p$ for *L. monocytogenes* bacteria, $Cu(Ac)_2.2H_2O$, $2a3NO_24mp$, 2a5Clp, $2a5NO_2p$ and **2** for E. *faecalis* bacteria, all compounds (except **3**) for *B. subtilis* bacteria, and $2a5NO_2p$ and **1** were observed for *C. albicans* yeast.

Keywords: 2-Methoxy-5-sulfamoylbenzoic acid, aminopyridine, Cu(II) metal complex, antifungal activity, antibacterial activity.

1. INTRODUCTION

Numerous pharmacological activities of 2-aminopyridines have been discovered, including antibacterial, antifungal, antihistaminic, analgesic, cardiotonic, antiviral, anticonvulsant, anti-diabetic, anti-Alzheimer, anti-parasitic and anti-inflammatory.[1] Even though the amino group participates in coordination in some investigations on 2-aminopyridine complexes.[2-4] Typically, 2-aminopyridines are monodentate and coordinate through the rings of nitrogen.[4,5]

The pharmacological effects of sulfamoylbenzoic acid derivatives, which include carbonic anhydrase and chorismate mutase inhibition, antibacterial, diuretic, anticonvulsant, and antihypertensive effects are recognized.[6-19] The salts[20-26] and mixed ligand metal complexes[27-30] with 2-aminopyridine derivatives of 2-methoxy-5-sulfamoylbenzoic acid (Hsba) and metal complex[31,32] of Hsba have all been created.

These days, it becomes ineffective because disease-causing microorganisms develop resistance to the medications used to treat them. Thus, novel compounds that may be acquired cheaply and effectively are required to eradicate hazardous microbes for human health. Future research will be aided by the discovery that the chemicals produced in this work exhibit antibacterial action against yeasts and bacteria. It has been demonstrated that co-crystals, salts, and metal complexes containing 2-methoxy-5-sulfamoylbenzoic acid and 2-aminopyridine derivatives can be used *in vitro* and *in vivo* antibacterial and antifungal experiments.[19-28,31,32]

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2. EXPERIMENTAL

2.1. General Methods and Materials

All of the compounds utilized were analytical reagents that were bought from Aldrich on the open market. Perkin Elmer Optima 4300 DV ICP-OES for AAS analysis, Bruker Optics Vertex 70 (in KBr) for FT-IR analysis, SHIMADZU UV-2550 (in DMSO, 10-3 M) for UV-Vis spectra analysis, Sherwood Scientific Magway MSB MK1 for magnetic susceptibility analysis and WTW Cond 315i/SET (in DMSO, 10-3 M) for molar conductivity analysis was used.

2.2. Synthesis of Metal Complexes (1-3).

A 1 mmol (0.2312 g) Hsba, 1 mmol (0.199 g) $Cu(CH_3COO)_2H_2O$ and 1 mmol 2-aminopyridine derivatives (0.1531 g $2a3NO_24mp$ for **1**, 0.1286 g 2a5Clp for **2** and 0.1391 g $2a5NO_2p$ for **3**) were dissolved in 50 mL of ethanol (50%). The mixture was stirred for a week at room temperature. The powdered green complexes precipitated in the reaction medium (82% yield for **1**, 75% yield for **2,** and 71% yield for **3**) were filtered and dried (Figure 1).

2.3. Antifungal and antimicrobial activity

The microbroth dilution method[33] was used to assess the antifungal and antimicrobial properties of Hsba, $2a3NO_24mp$, 2a5Clp, $2a5NO_2p$, $Cu(OAc)_2.2H_2O$, and complexes **1-3**. In this study, the antibacterial and antifungal activity of all the investigated compounds were investigated using *S. aureus*, *P. aeruginosa*, *E. Coli*, *B. subtilis*, *E.* faecalis, *L. monocytogenes* bacteria, and *C. albicans* yeast. Microorganisms *B. subtilis*, *S. aureus*, *L. monositogenes*, *P. aeruginosa*, and *C. albicans* were obtained from Technical University-Eskişehir; *E. coli* and *E. faecalis* were obtained from Osmangazi University-Eskişehir.

The compounds' antibacterial effectiveness was assessed using the microbroth dilution susceptibility test. The samples' stock solutions in DMSO were created. From 4 mg/mL to 0.007 mg/mL of sterile distilled water was used to construct dilution series, which were then transferred to 96-well microtiter plates. Using the McFarland No: 0.5 standard solution, bacterial and Candida albicans suspensions that had been cultivated overnight in double-strength Mueller-Hinton broth were standardized to 10^8 CFU/mL. Then, 100 µL of each suspension of the microorganisms was poured into each well. As the negative control, a well chain that had no microorganisms was employed. The medium and sterile distilled water acted as a positive growth control. The first well without turbidity was chosen as the lowest inhibitory concentration (MIC) after 18–24 hours of incubation at 37°. Fluconazole was employed as an antifungal drug, whereas Vancomycin, Cefepime, and Levofloxacin served as the conventional antibacterial agents. Table 1 lists the findings of the compounds and the pharmacological controls' antibacterial activity.

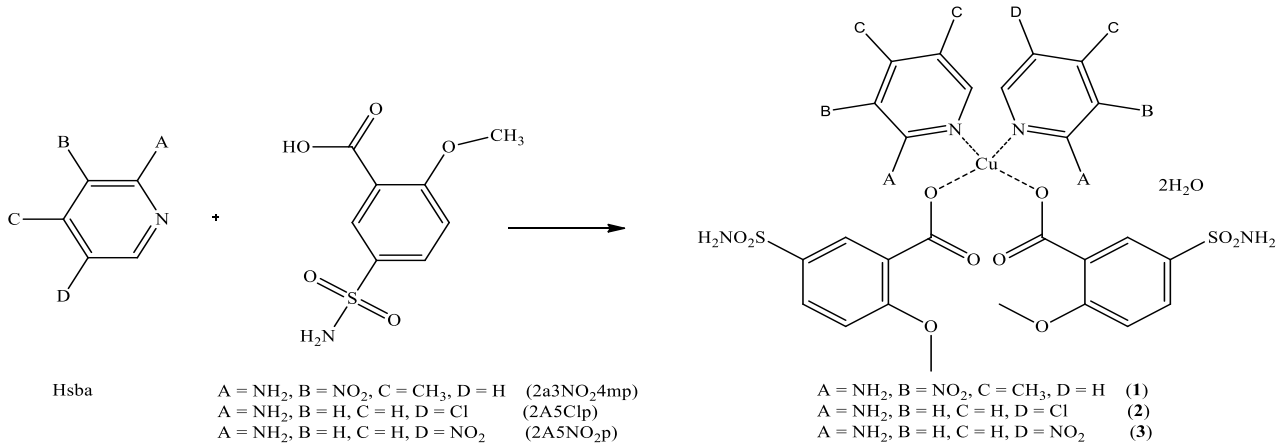

Figure 1. Syntheses of complexes **1-3**.

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3. RESULTS and DISCUSSION

3.1. Results of AAS

AAS results of the synthesized Cu(II) complexes {[M] %Found (Calcd.) 7.30(7.34) for **1**, for 7.80(7.78) **2** and 7.55(7.58) for **3**} Metal: Acid: Base ratios were found as 1:2:2. According to these results, it was observed that the experimentally obtained elemental analysis values were in agreement with the calculated theoretical elemental analysis values.

3.2. Results of IR Measurements

The IR data of all compounds are given in Table 1. The ν(OH) vibrations of water molecules in **1-3** were observed as broad absorption bands in the range 3581-3431 cm^{-1}. The absorption bands of NH_2 group 3364 and 3289 cm^{-1} for Hsba, 3451 and 3257 cm^{-1} for 2a3$NO_2$4mp, 3377 and 3223 cm^{-1} for 2a5Clp, 3458 and 3271 cm^{-1} for 2a5NO_2p are somewhat different from those found 3461, 3439, 3374 and 3318 cm^{-1} for **1**, 3469, 3328, 3266 and 3194 cm^{-1} for **2** and 3384, 3346, 3276 and 3238 cm^{-1} for **3** a result of the negligible intermolecular interactions. All compounds (except 2a5Clp and 2a5NO_2p) have faint bands in the range 2994-2760 cm^{-1} and 3114-3058 cm^{-1} which result from the stretching vibrations of the corresponding types of C-H, aromatic and aliphatic. The carboxylate groups the asymmetric (ν_{as}) and symmetric (ν_s) stretching vibrations at 1684 and 1480 cm^{-1} for H2MeO5sba, 1628 and 1429 cm^{-1} for **1**, 1640 and 1442 cm^{-1} for **2** and 1652 and 1461 cm^{-1} for **3**. The differences between the asymmetric and symmetric extension of the carboxylate groups of **1-3** (Δυ = 191-198) indicate that the carboxylate group bonds monodentate to the metal ion.[34] The strong absorption bands at the region of 1624-1375 cm^{-1}, 1557-1329 cm^{-1}, 1383-1090 cm^{-1}, 1278-1123 cm^{-1}, and 796-750 cm^{-1}, are attributed to the $\nu(NO_2)$ (expect Hsba, 2a5Clp and **2**), ν(C=C)/ν(C=N) (expect Hsba), ν(C-O) (expect aminopyridines), ν(S=O) (expect aminopyridines) and pyridine groups (expect Hsba), respectively. The weak bands at 577-572 cm^{-1} and 489-436 cm^{-1} are caused by the M-O and M-N vibrations of **1-3** (Table 1).

Table 1. Infrared band assignments for all compounds (cm^{-1}).

Assignment	Hsba	2a3$NO_2$4mp	2a5Clp	2a5NO_2p	1	2	3
ν(OH)	2900(br)	-	-	-	3478(br)	3581(br)	3431(br)
ν(NH_2)	3364(m)	3451(m)	3377(m)	3458(m)	3461(m)	3469(m)	3384(m)
	3289(m)	3257(m)	3223(m)	3271(m)	3439(m)	3328(m)	3346(m)
					3374(m)	3266(m)	3276(m)
					3318(m)	3194(m)	3238(m)
ν(C-H)$_{ar.}$	3110(s)	3114(w)	3058(w)	3086(w)	3083(w)	3075(w)	3085(w)
ν(C-H)$_{al.}$	2994(w)	2966(w)	-	-	2939(w)	2987(w)	-
	2954(w)	2886(w)			2879(w)	2900(w)	
	2850(w)	2848(w)			2760(w)	2852(w)	
ν(COO)	1684(s)	-	-	-	1628(s)	1640(s)	1652(s)
	1480(s)				1429(s)	1442(s)	1461(s)
ν(C=N)	1577(s)	1614(s)	1620(s)	1632(s)	1608(s)	1608(s)	1634(s)
ν(C=C)	1540(s)	1586(s)	1610(s)	1503(s)	1556(s)	1581(s)	1612(s)
	1493(s)	1505(s)	1585(s)	1427(s)	1522(s)	1561(s)	1580(s)
	1473(s)	1435(s)	1566(s)		1429(s)	1497(s)	1505(s)
	1436(s)		1477(s)			1467(s)	1484(s)
			1442(s)				
ν(NO_2)	-	1554(s)	-	1557(s)	1522(s)	-	1540(s)
		1374(s)		1329(s)	1354(s)		1344(s)
ν(C-O)	1356(s)	-	-	-	1383(s)	1375(s)	1382(s)
	1210(s)				1323(s)	1295(s)	1293(s)
	1050(s)				1093(s)	1091(s)	1090(s)
ν(S=O)	1277(s)	-	-		1278(s)	1277(s)	1270(s)
	1189(s)				1163(s)	1171(s)	1162(s)
	1149(s)				1123(s)	1125(s)	1120(s)
γ(py)	-	782(s)	784(s)	750(s)	784(s)	768(s)	796(s)
ν(M-N)	-		-		487(w)	436(w)	489(w)
ν(M-O)	-		-		577(w)	572(w)	577(w)

3.3. Results of UV/vis Spectrum

The electronic spectra of Hsba, 2a3$NO_2$4mp, 2a5Clp, 2a5NO_2p, and Cu(II) complexes (**1-3**) were recorded in DMSO solution with 1x10^{-3} $molL^{-1}$ concentrations. The π- π^* transitions of compounds are 302(32160) and 291(26530) nm for Hsba, 396(16810) and 292(29040) nm for 2a3$NO_2$4mp, 317(34750) nm for 2a5Clp, 359(33400) and 294(41190) nm for 2a5NO_2p, 295(35390) and 291(32490) nm for **1**, 309(43400) and

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302(48170) nm for **2** and 364(33260) and 295(14550) nm for **3**. The bands d-d transitions are observed at 791(230) nm for **1**, 770(320) nm for **2** and 780(200) for **3**.[30,35]

3.4. Results of Magnetic Susceptibility and Molar Conductivity.

Cu(II) complexes (**1-3**) have a 1.63 BM for, 1,64 BM for **2,** and 1.66 BM for **3** per metal ion magnetic moment at room temperature, which denotes the presence of one unpaired electron (Cu^{2+}, d^9). The compounds (**1-3**) are non-ionic complexes as evidenced by the molar conductivity values in DMSO, which are 8.10 $\Omega^{-1}cm^2mol^{-1}$ for **1**, 5.10 $\Omega^{-1}cm^2$ mol^{-1} for **2** and 2.20 $\Omega^{-1}cm^2$ mol^{-1} for **3**.[36]

3.5. Results of Antimicrobial Activity.

MIC (minimum inhibits concentration) values of $Cu(OAc)_2.2H_2O$, Hsba, $2a3NO_24mp$, 2a5Clp, $2a5NO_2p$, and Cu(II) complexes (**1-3**) were contrasted with the control drugs Vancomycin, Levofloxacin, Cefepime, and Fluconazole. Table 2 shows the results of antibacterial and antifungal activity of all substances and control drugs. According to the examination of those features, the compounds exhibit strong antibacterial activity. The MIC values for all strains that were tested are typically in the range of 7.80 and 250.00 µg/mL.

All antibacterial drugs and substances have activity against *E. coli;* **2** (7.80 µg/mL) showed higher action compared to what Vancomycin and Levofloxacin suggested while $2a5NO_2p$ showed similar effective (31.25 µg/mL). Other compounds were found to have a lower degree of according to Vancomycin and Levofloxacin {$Cu(Ac)_2.2H_2O$, $2a3NO_24mp$, **1** (62.50 µg/mL) > Hsba, 2a5Clp, **3** (125.00 µg/mL)}. **2** (7.80 µg/mL) and $2a5NO_2p$ (31.25 µg/mL) indicated greater activity than according to Cefepime while $Cu(Ac)_2.2H_2O$, $2a3NO_24mp$ and **1** showed equal effective (62.50 µg/mL). Hsba, 2a5Clp and **3** (125.00 µg/mL) seen lower degree of according to Cefepime. Higher action compared to what Vancomycin and Levofloxacin suggested.

S. aureus; $Cu(Ac)_2.2H_2O$ (31.25 µg/mL) showed similar activity compared to what Vancomycin and Levofloxacin while other compounds saw lower degree of activity {$2a3NO_24mp$, 2a5Clp, $2a5NO_2p$, **1** (62.50 µg/mL) > **2**, Hsba (125.00 µg/mL) > **3** (250.00 µg/mL)}. $Cu(Ac)_2.2H_2O$ (31.25 µg/mL) indicated greater activity than according to Cefepime while $2a3NO_24mp$, 2a5Clp, $2a5NO_2p$, and **1** showed equally effective (31.25 µg/mL). Compound **2**, Hsba (125.00 µg/mL), and **3** (250.00 µg/mL) were found to have a lower degree of according to Cefepime.

Table 2. Activity values (µg/mL) of all compounds

Compound	*E. coli*	*S. aureus*	*E. faecalis*	*P. aeruginoa*	*L. monocytogens*	*B. subtilis*	*C.albicans*
Cefepime	62.50	62.50	31.25	31.25	31.25	62.50	-
Levofloxacin	31.25	31.25	62.50	31.25	31.25	62.50	-
Vancomycin	31.25	31.25	62.50	62.50	125.00	250.00	-
Fluconazole	-	-	-	-	-	-	62.50
$Cu(Ac)_2.2H_2O$	62.50	31.25	62.50	62.50	62.50	62.50	125.00
Hsba	125.00	125.00	125.00	31.25	31.25	62.50	125.00
$2a3NO_24mp$	62.50	62.50	62.50	62.50	31.25	62.50	125.00
2a5Clp	125.00	62.50	62.50	62.50	125.00	62.50	125.00
$2a5NO_2p$	31.25	62.50	62.50	62.50	31.25	62.50	62.50
1	62.50	62.50	125.00	62.50	62.50	62.50	62.50
2	7.80	125.00	62.50	31.25	62.50	62.50	125.00
3	125.00	250.00	250.00	250.00	250.00	125.00	250.00

E. faecalis; all compounds showed less activity than compared to what Cefepime {Cefepime (31.25 µg/mL) > $Cu(Ac)_2.2H_2O$, $2a3NO_24mp$, 2a5Clp, $2a5NO_2p$, **2** (62.50 µg/mL) > **1**, Hsba (125.00 µg/mL) > **3** (250.00 µg/mL)}. $Cu(Ac)_2.2H_2O$, $2a3NO_24mp$, 2a5Clp, $2a5NO_2p$, and **2** (62.50 µg/mL) observed similar activity according to Vancomycin and Levofloxacin while other compounds seen lower degree activity {**1**, Hsba (125.00 µg/mL) > **3** (250.00 µg/mL)}.

P. aeruginoa; Hsba and **2** (31.25 µg/mL) showed greater activity than according to Vancomycin while $Cu(Ac)_2.2H_2O$, $2a3NO_24mp$, 2a5Clp, $2a5NO_2p$ and **1** equally effective (62.50 µg/mL). Compound **2** (250.00 µg/mL) was found to have a lower degree of according to Vancomycin. Hsba and **2** (31.25 µg/mL) showed similar activity compared to what Cefepime and Levofloxacin while other compounds saw lower degree activity {$Cu(Ac)_2.2H_2O$, $2a3NO_24mp$, 2a5Clp, $2a5NO_2p$, **1** (62.50 µg/mL) > **2** (250.00 µg/mL)}.

L. monocytogens; when MIC values are compared; Hsba, $2a3NO_24mp$, $2a5NO_2p$ (31.25 µg/mL) and $Cu(Ac)_2.2H_2O$, **1**, **2** (62.50 µg/mL) indicated greater activity than according to Vancomycin while 2a5Clp showed equally effective (125.00 µg/mL). Compound **3** (250.00 µg/mL) was found to have a lower degree of according to Vancomycin. Hsba, $2a3NO_24mp$, $2a5NO_2p$

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(31.25 µg/mL) showed similar activity compared to what Cefepime and Levofloxacin while other compounds saw lower degree activity {$Cu(Ac)_2.2H_2O$, **1**, **2** (62.50 µg/mL) > 2a5Clp (125.00 µg/mL) > **3** (250.00 µg/mL)}.

B. subtilis; all compounds showed greater activity than according to Vancomycin (250.00 µg/mL) {Hsba, $Cu(Ac)_2.2H_2O$, $2a3NO_24mp$, 2a5Clp, $2a5NO_2p$, **1**, **2** (62.50 µg/mL) > **3** (125.00 µg/mL)}. Hsba, $Cu(Ac)_2.2H_2O$, $2a3NO_24mp$, 2a5Clp, $2a5NO_2p$, **1**, **2** (62.50 µg/mL) showed similar activity compared to what Levofloxacin and Cefepime while **3** seen lower degree activity (125.00 µg/mL).

The antifungal drug and substances have activity against *C. parapisilosis* when MIC values are compared; $2a5NO_2p$ and **1** (62.50 µg/mL) observed similar activity according to Fluconazole while the other compounds seen less level of activity (125.00 µg/mL).

4. CONCLUSION

This work is the first to create three mixed ligand copper(II) complexes (**1-3**) of 2-aminopyridine derivatives and 2- methoxy-5-sulfamoyl benzoic acid. Spectroscopic examination results point to the possibility of tetrahedral complexes being formed from the formulae of **1-3**. The metal:acid: base ratio was 1:2:2 for **1-3**. Each substance shows action against yeast and bacteria. $Cu(Ac)_2.2H_2O$, $2a3NO_24mp$, 2a5Clp, $2a5NO_2p$, **2** (62.50 µg/mL) for *E. faecalis*, Hsba, $2a3NO_24mp$ and $2a5NO_2p$ (31.25 µg/mL) for *L. monocytogens*, Hsba, $Cu(Ac)_2.2H_2O$, $2a3NO_24mp$, 2a5Clp, $2a5NO_2p$, **1** and **2** (62.50 µg/mL) for *B. subtilis*, $Cu(Ac)_2.2H_2O$ (31.25 µg/mL) for *S. aureus*, **2** (7.80 µg/mL) for *E. Coli*, Hsba and **1** (31.25 µg/mL) for *P. aeruginoa*, $2a5NO_2p$ and **1** (62.50 µg/mL) for *C. albicans* have the best activity. A number of human diseases are brought on by certain fungal and bacterial strains, which are among the most prevalent pathogens. So, it is possible to analyze these produced compounds (**1-3**) for the manufacture of new antibacterials.

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Conflict of Interest

The authors declare that there is no conflict of interest with any person, institute, company, etc.

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