

Synthesis of (3,4-bis{[2-hydroxy-3-methoxy-5-(4-methylphenyl azo)benzylidene]-amino}phenyl phenyl methanone as a novel azo Schiff base

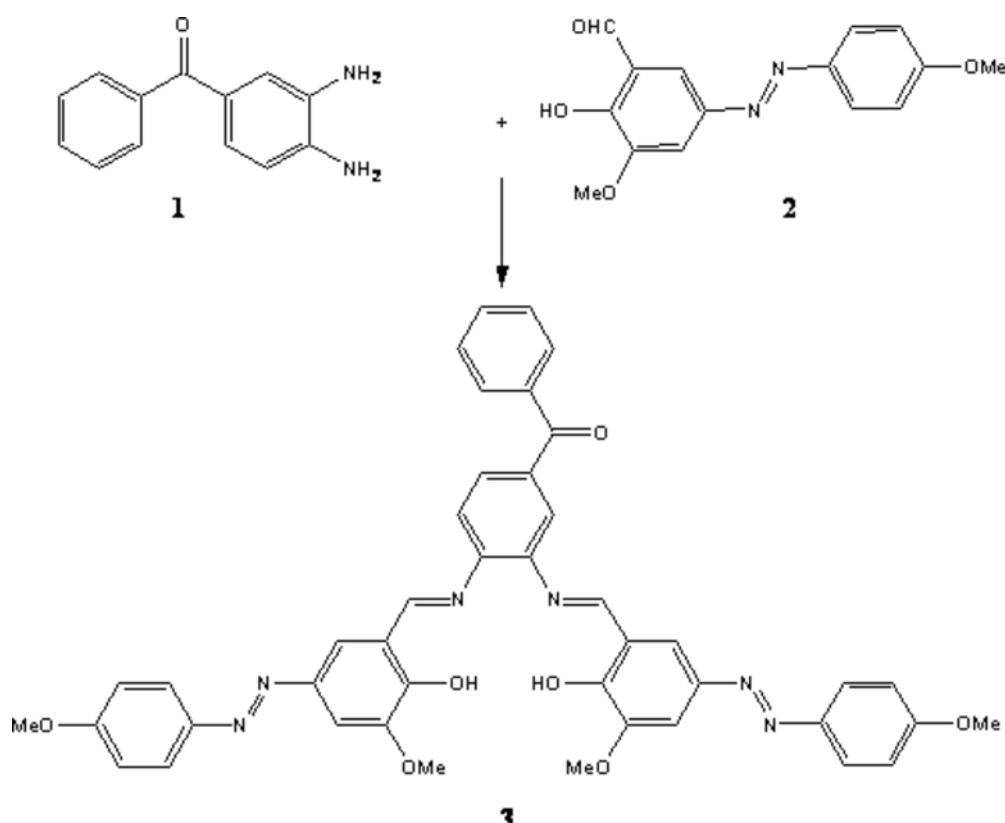
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Schiff bases are used as substrates in the preparation of a large of bioactive and industrial compounds via ring closure, cycloaddition, replacement reactions, etc [1]. In additon, Schiff bases are well known to have biological activites such as antibacterial [2-5], antifungal [4-6], antitumor [7-9], and herbicidal activities [10]. Also Schiff bases are used as ligands for complexation of some metal ion [11]. Azo compounds are widely used as dyes and pigments [12]. Another area of application of aromatic azo compounds is analytical chemistry where some of these compounds are used as indicators in pH, redox or complexometric titrations [13-14]. On the other hand azo compounds show biological activities containing antibacterial [15-16], pesticidal [10] activities. According to above facts, we decided to synthesize a new azo Schiff base **3** as potential biological and complexometric agent. Its biological activities and its uses in analytical works are under study.



Azo aldehyde **2** was synthesized according to a reported method [17]. To a stirred solution of azo aldehyde **2** (1.08 g, 3.76 mmol) in dry CH₂Cl₂ (30.00 mL) at 0 °C were successively added (3,4-diaminophenyl)phenyl methanone **1**(0.40 g, 1.88 mmol) and a excess of anhydrous MgSO₄ (2.00 g, 16.67 mmol). The resulting mixture was stirred for 6 hours at room temperature [18]. The mixture was filtered and washed with dichloromethane. Then the solvent was evaporated under reduced pressure to give azo Schiff base **3** as a red solid which was recrystallized from ethanol 95% (1.28 g, 91 %)

m.p. 140-142 °C.

IR (KBr) (cm⁻¹): 1604.7 (C=N), 1658.9 (C=O), 3155.3-3649.1 (OH).

¹H-NMR (CDCl₃) (250 MHz) δ(ppm): 3.91 (2 OMe, s, 6H), 3.98 (2 OMe, s, 6H) 6.89-7.01 (C₆H₅COC₆H₃, m, 8H), 7.24-7.85 (MeOC₆H₄N₂C₆H₂(OH)(OMe), m, 12H), 8.70 (2HC=N, s, 2H), 13.89 (2OH, br, 2H).

¹³C-NMR (CDCl₃) (62.90 MHz) δ(ppm): 56.43, 56.55, 105.73, 114.40, 114.73, 114.13, 118.67, 122.41, 123.13, 124.67, 127.82, 128.65, 130.37, 132.16, 132.40, 133.65, 138.86, 146.00, 147.10, 149.54, 154.23

MS (m/z, %): 492 (C₆H₅COC₆H₃N=CC₆H₃OHOMeN=CC₆H₃OHOMeN, 3.6), 478 (C₆H₅COC₆H₃ (N=C C₆H₃OHOMe)₂, 31.9), 464 (C₆H₅COC₆H₃N=CC₆H₃OHOMeN=NC₆H₄OMe, 2.2), 343 (C₆H₅CO C₆H₃N=CC₆H₃OHOMeN, 11.8), 135 (MeOC₆H₄N=N, 10.7), 108 (MeOC₆H₅, 100.0), 105 (C₆H₅CO, 42.3), 77 (C₆H₅, 71.8).

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