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Universal Journal of Pharmaceutical Research
An International Peer Reviewed Journal
ISSN: 2831-5235 (Print); 2456-8058 (Electronic)

RESEARCH ARTICLE

STUDY OF SOME FLUOREN-9-ONE THIOSEMICARBAZONES: SYNTHESIS, CATALYTIC EFFECTS AND SPECTRAL CHARACTERIZATION

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Article Info:

Article History:
Received: 15 July 2024
Reviewed: 10 September 2024
Accepted: 23 October 2024
Published: 15 November 2024

Cite this article:
GLINMA B, KPADONOU B, AGNIMONHAN H, MEDEGAN S, KPOVIESSI S, KAPANDA C, GBAGUIDI F. Study of some fluoren-9-one thiosemi-carbazones: Synthesis, catalytic effects and spectral characterization. Universal Journal of Pharmaceutical Research 2024; 9(5): 30-34.
http://doi.org/10.22270/ujpr.v9i5.1194

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Abstract

Aim and objective: Nowadays, Schiff bases have shown a remarkable importance in medicinal chemistry research. Semicarbazones, thiosemicarbazones and their derivatives are important Schiff base ligands with biological properties. Fluorenones are important organic compounds used in many different fields. Here, we reported the synthesis of fluoren-9-one thiosemicarbazones (F1-F4).
Methods: For their synthesis, we first carried out a theoretical study based on the physical properties (reasonable pharmacokinetics and drug availability) of the compounds using Lipinski's rules. We also varied the reaction conditions using several reagents (HCl, 1N; H_2SO_4 conc. and GAA) and ethanol in order to optimize reaction yields. Structure of each molecule was characterized by spectrometrical analysis (MS and IR, and especially NMR ^1H and ^{13}C).
Results: During the course of the study, we observed an increase in reaction yields in the presence of the less aqueous acids, particularly with GAA, which gave the best yields (89, 77, 91 and 96%) for molecules F1, F2, F3 and F4 respectively.
Conclusion: We noted that compounds presented essential properties to exhibit pharmaceutical activities. They could have a variety of pharmaceutical activities on pathogens resistant to existing treatments.
Keywords: Catalyst, fluoren-9-one, pharmaceutical properties, synthesis.

INTRODUCTION

Fluoren-9-one is a versatile compound with a range of properties. It widely used for preparing dye material and modifying resin and other materials[1,2]. A fluorene derivative, this compound is used in antimalarial drugs synthesis. It is used in polymers and dyes too. Fluorenone is used in organic electronic material synthesis. Some examples: (i) blue and green phosphorescent organic LED host synthesis; (ii) synthesising fluorene-based molecular motors; (iii) synthesis of chichibabin hydrocarbons for organic spintronics; (vi) it is also involved in the formation of picenes through the photosensitisation reactions of 1,2-di(1-naphthyl)ethane[3]. Fluorenone is used to manufacture pharmaceuticals, particularly antimalarial drugs. Fluorenone substitutes act as antibiotics, cancer treatments, antivirals and nerve agents[4]. Some substituted 'azafluorenones are biologically active, such as the naturally occurring antimicrobial compound onychine (1-methyl-4-azafluorenone)[5]. 1,8-Diazafluoren-9-one is used for fingerprint detection. Thiosemicarbazones are nitrogen- and sulphur-containing compounds used in synthesis. They are sulfur-based derivatives of semicarbazones[6]. They derivative molecules are organic molecules that are of great biological importance[6-9]. Their structure allows for flexible bonds[10]. They have a wide range of applications in materials science. The chemistry of thiosemicarbazones has been researched for 20 years, and fluorescent derivatives have been reported[11,12]. Many studies have been conducted on the structure-activity relationship of thiosemicarbazones. Changes in the amino substituents or N-heterocyclic ring affect biological activity. Changes in the aldehyde or ketone structure also affect antibacterial and anticancer

activity[13,14]. With all its beneficial properties cited in the literature, and with the aim of bringing added value to the synthesis, we initiated the synthesis and study of catalysts on the yield of reactions between fluoren-9-one and thiosemicarbazides used to have new organic molecules of pharmacological interest.

MATERIALS AND METHODS

In current work, reactants were directly used without any further purification. Obtained from chemical companies including Sigma-Aldrich, Acros Organic, Janssen Chimica, Supelco and Riedel-de Haen, they are fluoren-9-one (98%), hydrochloric acid HCl (37%, 1N), sulfuric acid H_2SO_4 (95-97%), and glacial acetic acid GAA (≥ 99%), technical ethanol (EtOH, 96°), thiosemicarbazide and derivatives 2-methyl-, 4-methyl- and 4-phenyl-3-thiosemicarbazide (99.5%). Products obtained from the synthesis were characterised by elemental analyses such as: (i) Proton and carbon-13 NMR spectra (on a Bruker Avance 400 UltraSheild apparatus with DMSO-D_6 or $CDCl_3$ as solvent) were recorded when the instrument was calibrated to frequencies of 400.130 MHz and 100.612 MHz for proton and ^{13}C respectively. Chemical shifts (δ) are expressed in ppm relative to the reference tetramethylsilane. The multiplicity of ^1H NMR signals has been classified: singlet (s), doublet (d), triplet (t), multiplet or massif (m) etc. (ii) Mass spectra were recorded in positive mode and by atmospheric pressure chemical ionization (APCI), and masses are given in m/z ([MH^+]). The melting points (p.m.) of each product has not been determined with our "Köfler bench" apparatus.

To synthesize the different molecules (thiosemi-carbazones, F1-F4), we explored the methods described in the previous work (Figure 1), varying the reaction catalysts[15,16], and then they have been characterized by spectrometrical analysis methods.

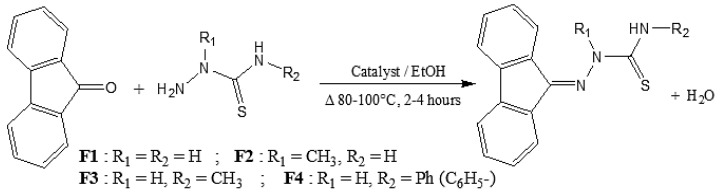

Figure 1: Compound synthesis reaction.

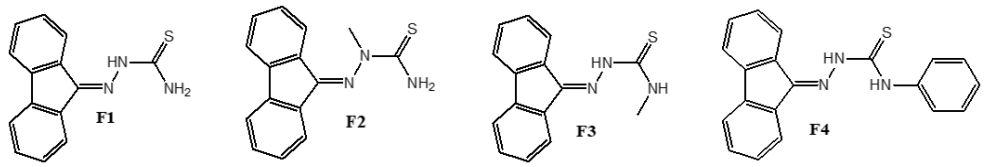

Figure 2: Structure of synthesized compounds.

General method: In an Erlenmeyer, prepare in a solution of 0.01 mole of fluoren-9-one dissolved in 10-20 mL of ethanol with 1 mL of HCl (1N) or a few drops (maximum 3-4) of concentrated H_2SO_4 or 1 mL of glacial acetic acid (GAA). In a flask, prepare a solution (0.01 mole) of thiosemicarbazide or derivatives in 10 mL of ethanol, and then make the mixture by adding fluoren-9-one solution to thiosemicarbazide solution slowly. This mixture was refluxed for 2 to 4 hours with magnetic stirring. After cooling the reaction mixture, the precipitate obtained was filtered and washed with fresh distilled water until neutrality. The crude product was dried and recrystallised in ethanol to yield the purified molecule.

RESULTS AND DISCUSSION

Four products have been synthesized: fluoren-9-one thiosemicarbazone (F1), fluoren-9-one 2-methyl-3-thiosemicarbazone (F2), fluoren-9-one 4-methyl-3-thiosemicarbazone (F3) et fluoren-9-one 4-phenyl-3-thiosemicarbazone (F4). We checked the chemical structure of the predefined molecules to obtain the correct structural information.[17]

The scaffold is low molecular weight, reasonably lipophilic (Log P or C log P), capable of forming hydrogen bonds (Figure 2, Table 1) and can be synthesized economically[17,18,19].

Table 1: Physical Properties Compatible with Reasonable Pharmacokinetics and Drug Availability of synthesized molecules.

Compound	Molecular weight (g.mol^{-1})	*LogP*[17]	*Clog P*[19]	Number of H bond donors	Number of H bond acceptors	Number of criteria met
Rules	< 500	< 5		≤ 5	< 10	at least 3
F1	253.3222	2.8	3.57	3	3	all
F2	267.3488	3.04	2.46	2	3	all
F3	267.3488	3.32	4.05	2	3	all
F4	329.4182	4.99	5.38	2	3	all / 3

Table 2: Connolly parameters (solubility and stability) of compounds at 298°K.

Compound	Connolly Accessible Area (A^2)	Connolly Molecular Area (A^2)	Connolly Solvent-Excluded Volume (A^3)
F1	389.468	234.517	187.501
F2	404.675	247.389	204.694
F3	412.998	252.928	207.703
F4	507.653	313.174	255.141

The more data available for modelling, the more reliable the results[17]. Analyzing the results of a few physical parameters according to QSAR study, we note that all the compounds met the criteria of bioavailability and pharmacokinetics, essential properties for presenting pharmaceutical activities. In addition, all our compounds have good lipophilicity, Log P<5, which is an important feature in pharmacological studies. This term reflects a substance's ability to enter cells through lipid membranes, indicating toxicant uptake and baseline toxicity[17].

Table 3: Catalyst dependent variation in synthesis yield.

	Yield of compounds in %			
Catalyst	**F1**	**F2**	**F3**	**F4**
HCl, 1N	76.37	65.15	71.25	77.27
H_2SO_4 conc	85.24	71.35	75.89	88.68
GAA	89.51	77.43	91.74	96.39

During the work, studies (from Chem3D Ultra 8.0) on solubility and stability in subcritical water were reported (Table 2). Green solvent data on solubility and degradation of organic compounds is needed for environmental remediation, chemistry, chemical engineering, medicine and more. The solubility of organic compounds increases with water temperature[20]. The lipophilicity agree well with the surface areas and exchange volumes obtained from theoretical calculations. The more lipophilic F4 liquid product has the widest distribution of available surface area and volume, and therefore the greatest solubility. Find it in the literature, studies showed that a molecular dot surface is a smooth envelope of points on the molecular surface. Organic compound solubilities affect process equipment design and operation. The free volume and distribution of the compound will affect the penetrants transport properties. Low-polarity organics are insoluble in water at ambient conditions, but at higher temperatures, they become soluble[21].

In the course of our study we investigated the effect of catalysts. Results have been shown in Table 3. Among the compounds, 4-phenyl-3-thiosemicarbazone (F4) gave the highest yields. Overall, it was observed that the F2 product gave the lowest yield each time with the variation of catalysts. This could be due to steric effects brought about by methyl on the nitrogen atom (N2). We noticed that the stronger and less aqueous the acid used, the higher the yield. As the synthesis is a condensation reaction leading to an imine function, the stronger the nucleophile of the acid used, the more it could still react on the product obtained. Hydrochloric acid being aqueous, the reaction is slow and leads to low yields, while concentrated sulfuric acid and GAA (both less aqueous) improved the reaction yield. This result confirmed one of our previous studies in which less aqueous reaction media favoured the yield of products obtained[22].

Structural characterization of synthesized compounds

Fluoren-9-one thiosemicarbazone (**F1**).

Yield: 76-89%.

^{1}H NMR with DMSO-d_6 as solvent and δ expressed in ppm: from 6.04 to 6.45 (broad s, 2H); 7.30-7.62 (m, $8H_{-Ar}$); 8.04 (s, 1H, N^2-H).

^{13}C NMR with DMSO-d_6 as solvent and δ expressed in ppm: 185.17 C=S, 157.36 C=N, 144.21, 132.17, 130.31, 130.07, 129.55, 128.63.

Mass by **LC-MS** (MH^+): 254.34. Theoretical formula: $C_{14}H_{11}N_3S$.

Fluoren-9-one 2-methyl-3-thiosemicarbazone (**F2**).

Yield: 65-77%.

^{1}H NMR with $CDCl_3$ as solvent and δ expressed in ppm: 6.57 (s, 2H, $-NH_2$); 7.33-7.73 (m, $8H_{-Ar}$); 3.16 (s, 3H).

^{13}C NMR with $CDCl_3$ and as solvent and δ expressed in ppm: 176.31 C=S, 156.65 C=N, 144.21, 133.15, 130.31, 130.17, 129.47, 128.77 ($C_{-Ar\ fluorenyl}$), 44.27 (^2N-CH_3).

Mass by **LC-MS** (MH^+): 268.35. Theoretical formula: $C_{15}H_{13}N_3S$.

Fluoren-9-one 4-methyl-3-thiosemicarbazone (**F3**).

Yield: 71-91%.

^{1}H NMR with DMSO-d_6 as solvent and δ expressed in ppm: 7.33-7.73 (m, $8H_{-Ar}$); 2.96 (s, 3H); 7.85 (s, 1H, N^4-H); 8.97 (s, 1H, N^2-H).

^{13}C NMR with DMSO-d_6 as solvent and δ expressed in ppm: 183.21 C=S, 159.23 C=N, 143.51, 132.16, 131.05, 130.23, 129.57, 128.67 ($C_{-Ar\ fluorenyl}$), 33.35 (^4N-CH_3).

Mass by **LC-MS** (MH^+): 268.33. Theoretical formula: $C_{15}H_{13}N_3S$.

Fluoren-9-one 4-phenyl-3-thiosemicarbazone (**F4**).

Yield: 77-96%.

^{1}H NMR with DMSO-d_6 as solvent and δ expressed in ppm: 7.39-7.67 (m, $8H_{-Ar}$); 6.51-7.23 (m, $5H_{-Ar}$); 8.35 (s, 1H, N^4-H); 9.27 (s, 1H, N^2-H).

^{13}C NMR with DMSO-d_6 as solvent and δ expressed in ppm: 187.35 C=S, 154.74 C=N, 143.51, 132.16, 131.05, 130.23, 129.57, 128.67 ($C_{-Ar\ fluorenyl}$); 137.54, 129.21, 126.72, 125.23 ($C_{-Ar\ phenyl}$).

Mass by **LC-MS** (MH^+): 330.43. Theoretical formula: $C_{20}H_{15}N_3S$.

For structural analysis of the compounds, we used their LC/MS spectra and, above all, proton and carbon-13 NMR. From these spectra, we obtained the molar mass, signals and peaks characteristic of the different products obtained. The ^{1}H and ^{13}C NMR spectra showed signals and peaks characteristic of protons and

carbons present in the structure of each molecule, such as fluorenone aromatic proton groups (chemical shift δ in ppm) ranging from 7.30 to 7.73 ($8H_{Ar}$); $-NH_2$ protons at around "broad" 6.04-6.45 ppm (F1) and 6.57 ppm (F2) because the two Hs have different chemical environments; $-N^2H-$ protons at 8.04, 8.97 and 9.27 ppm for F1, F3 and F4 respectively. This observed difference is due to the chemical environment and electronic effects within each structure. We also noted the presence of characteristic signals at 7.85 and 8.35 respectively from $-N^4H-$ for products F4 and F5. Signals from the N-substituted methyl protons of compounds F2 and F3 were also obtained at somewhat weaker fields than classical methyl, as they were located on nitrogen atoms at various electronic effects (3.16 and 2.96 ppm respectively). F4 showed signals corresponding to N^4-substituted phenyl. In ^{13}C NMR, we focused more on the peak of the imine carbon C=N, the main function characterizing the product of the condensation reaction. Chemical shift peaks between 154.74 and 159.23 ppm correspond to C=N in our products. We observed the disappearance of the carbonyl peak (C=O) previously present in the fluorenone structure, at a chemical shift of 201 ppm. The other structural elements of each molecule remain virtually identical to their respective base spectra (C=S, C_{Ar}, $-CH_3$, C-phenyl). LC/MS spectra, obtained in positive mode, gave us the molecular weights of each compound [MH^+]. These analyses revealed the purity of our synthesized products. This work in agreement with those reported[8,10,16,22-26].

Limitations of the study

During the course of current study, there was problems with the cold dissolution of the reactants. There was need to heat the mixture at 60°C to obtain a clear solution.

CONCLUSIONS

The synthesis of four organic molecules with the catalyst effects has been reported The scaffold has advantageous properties. In the catalyst study, it showed that the stronger and less aqueous the acid used, the higher the yield. All the compounds met the criteria of bioavailability and pharmacokinetics, their solubility was significantly enhanced with increasing water temperature, essential properties for presenting pharmaceutical activities.

ACKNOWLEDGEMENTS

We would like to thank Prof. Jacques POUPAERT and Prof. Didier LAMBERT for their contribution to the recording of spectra in their research unit, School of Pharmacy at the LDRI Institute in Belgium.

AUTHOR'S CONTRIBUTION

Glinma B: design, methodology, synthesis, review. **Kpadonou B**: writing original draft of article and comments, . **Agnimonhan F**: spectral data processing and comments, co-writer of the original draft. **Medegan S** and **Kpoviessi S**: review and editing. **Kapanda C**: recording of product spectra. **Ggaguidi F**: research supervisor. Final article was checked and approved by all authors.

DATA AVAILABILITY

The accompanying author can provide the empirical data that were utilized to support the study's conclusions upon request.

CONFLICT OF INTEREST

None to declare.

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