

STUDYING OF THERMAL, CHROMATOGRAPHIC, CHEMICAL, MICROBIAL-BEHAVIOR OF (SULFUR AND NITROGEN)-ORGANIC COMPOUNDS

**NAGHAM MAHMOOD ALJAMALI*, FAEZ ABDULHUSSEIN ALRAMMAHI, RADHIYA A KHDUR,
SAHER MAHMOOD JAWD**

Faculty of Education for Women, Kufa University, Iraq. Email: Dr.Naghm_mj@yahoo.com

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ABSTRACT

Objective: Series sulfur and nitrogen organic compounds were prepared in our previous papers in the first reference, from present studying which act various compounds (thia, sulfur, aze, and sulfide) from halogenation reaction, condensation reaction, and cyclization reaction.

Methods: The synthesized compounds have been studied (chromatography behavior, differential scanning calorimetry [DSC] - thermal measurements) for stability of compounds, studying of physical and other analytical studies such as solubility in various solvents, then tested in microbial studying.

Results: All synthesized compounds appeared high stability in thermal measurements and gave good evidence for clear separation for all tested compounds.

Conclusion: The prepared compounds have a high activity which due to the presence of nitrogen and sulfur atoms in their structures.

Keywords: Solute, Skin, Hospital.

INTRODUCTION

Sulfur and nitrogen organic compounds are important glass in synthetic chemistry, which act saturated and unsaturated compounds, often in combination with other heteroatoms such as thiiranes, thiirenes, thietanes, thietes, dithietanes, thiolanes, thianes, dithianes, thiepanes, thiippines, thiazoles, and thiophenes. Organic compounds of sulfur act responsible for the many of the unpleasant odors from decaying organic matter. Sulfonamide drugs were the first antibiotics in pharmaceutical chemistry and paved the way for the antibiotic revolution in medical field [1-6] (Scheme 1a and b).

Sulfur atom is a part of several sulfa drugs; it is the basis of many groups of drugs. The original antibacterial sulfonamides are synthetic antimicrobial agents that contain the sulfonamide group. Some sulfonamides are also devoid of antimicrobial activity like the anticonvulsant [7-10] (Scheme 2a and b).

Chromatography technique was first employed in Russia by the Italian-born scientist Mikhail Tsvet in 1900. He continued to work with chromatography in the first decade of the 20th century, primarily for the separation of plant pigments such as chlorophyll, carotenes, and xanthophylls. These components have various colors (green, orange, and yellow, respectively); they gave the technique its name. New types of chromatography developed during the 1930s and 1940s made the technique useful for many separation processes.

Chromatography technique developed substantially as a result of the work of Archer John Porter Martin and Richard Laurence Millington Synge during the 1940s and 1950s, for which they won a Nobel prize. They established the principles and basic techniques of partition chromatography and their work encouraged the rapid development of several chromatographic methods [11-17]. Column chromatography technique is a separation technique, in which the stationary bed is within a tube. The particles of the solid stationary phase or the support coated with a liquid stationary phase may fill the whole inside volume of the tube (packed column) or be concentrated on or along the inside tube wall leaving an open, unrestricted path for the mobile phase in the middle part of the tube (open tubular column). Differences in rates of movement through the medium are calculated to different retention times of the sample [15-17].

MATERIALS AND PROCEDURES

Differential scanning calorimetry (DSC) - thermal analysis carried out in Canada; chromatographic analysis in Canada; physical and analytical studies with the chemical and biological materials like Agar for bacteria and some instrumentals carried out in college of education; biological activity carried out in bio-lab in biodepartment; three types of bacteria collected from samples of biodepartment.

Prepared compounds in our previously work [1]

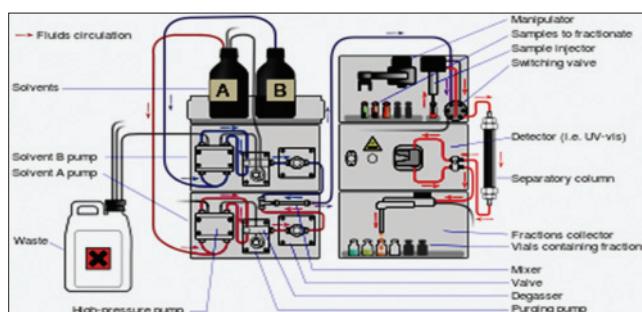
In our past work, we prepared several (sulfur and nitrogen)-organic compounds in the following schemes, but we will study the microbial activity for them on patients from hospital, the compounds in this work (Scheme 3 and 4).

Behavior of compounds in chromatography technique

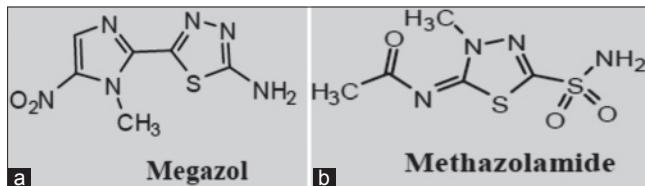
Analysis of some compounds in chromatography [14]

To studying of chromatography behavior, several solutions of compounds [4,8,11-13] were prepared via injection method through system to separation of selected compounds, all results of this studying in Figs 1-5.

All selected compounds separated according to infinity and polarity of terminal groups in compounds



The compounds will spend most of time migrating with the stream and will be transported away from other molecules which retained longer by the stationary phase. The ratio of the times spent in the moving and stationary regions is equal to the ratio of its concentrations in these regions, known as the partition coefficient. The term adsorption isotherm is often used when a solid phase is involved. The driving force for solute migration is the moving fluids, and the resistive force is the solute affinity for the stationary phase; the combination of these forces, as manipulated by the analyst, produces the separation [12,14-16].



Scheme 1: Sulfur and nitrogen drugs

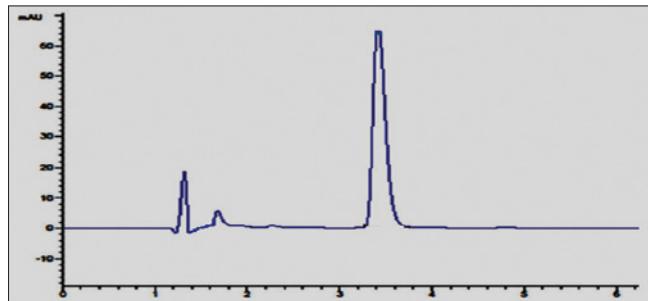
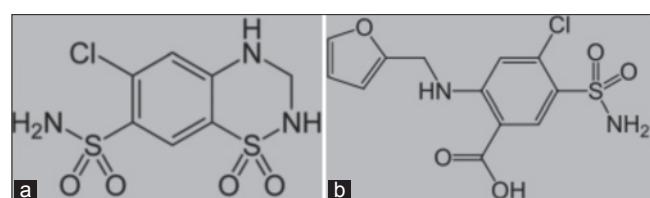


Fig. 1: Chromatogram of compound (4)



Scheme 2: Sulphanamide drugs

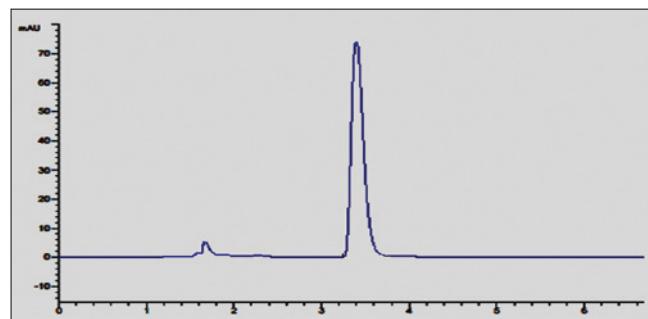
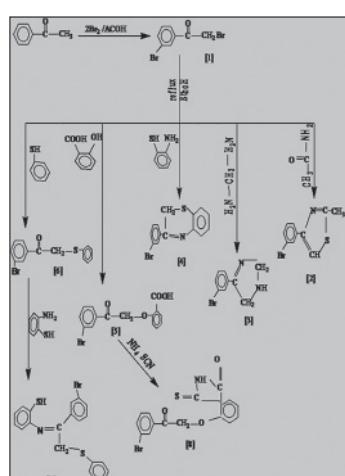


Fig. 2: Chromatogram of compound (8)



Scheme 3: Compounds [1-8]

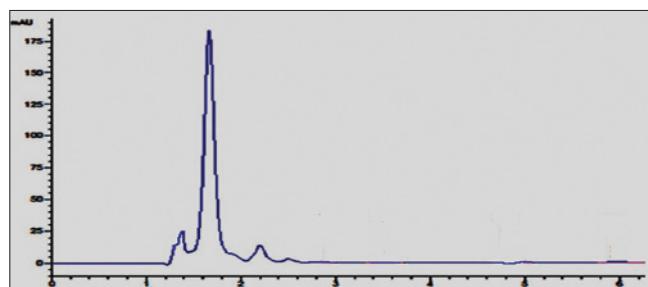
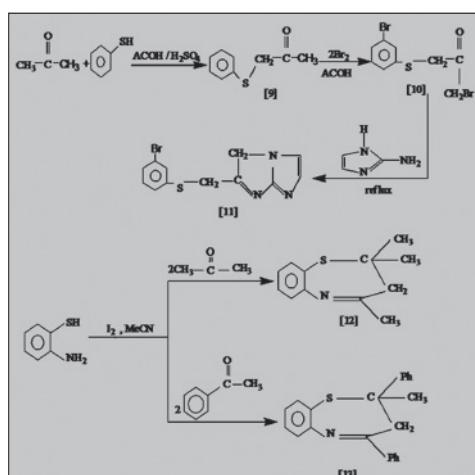


Fig. 3: Chromatogram of compound (11)



Scheme 4: Compounds [9-13]

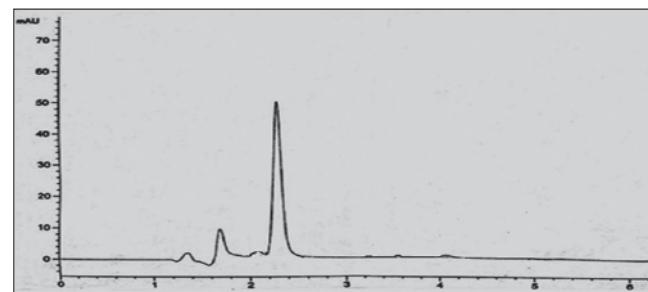


Fig. 4: Chromatogram of compound (12)

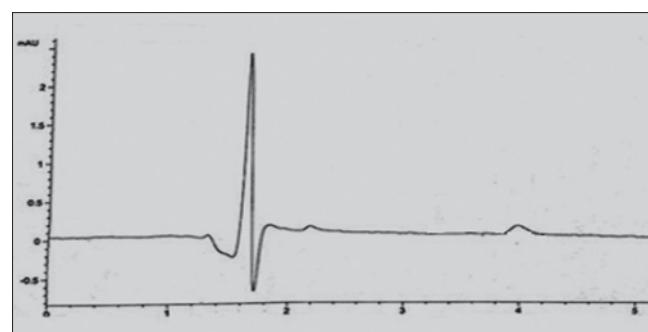


Fig. 5: Chromatogram of compound (13)

The results indicate that the selected compounds separated according to molecular weight and interaction between active groups in the compounds in separation column of chromatography technique.

Studying of thermal behavior (DSC-measurements)

DSC-measurements of some compounds measured for stability of sulfur and nitrogen compounds in Fig. 6-10, DSC-curves showed high stability [14] toward high temperature.

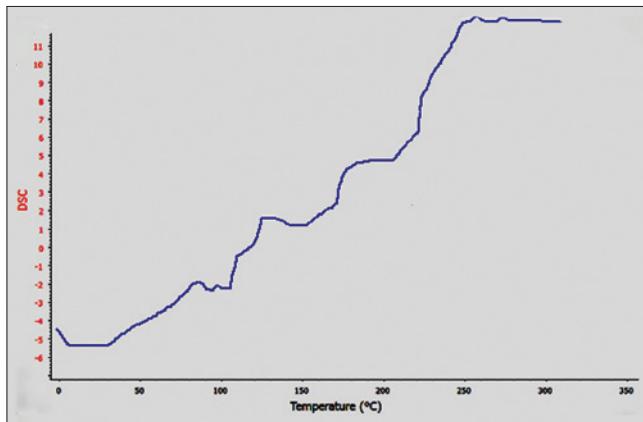


Fig. 6: Differential scanning calorimetry of compound (2)

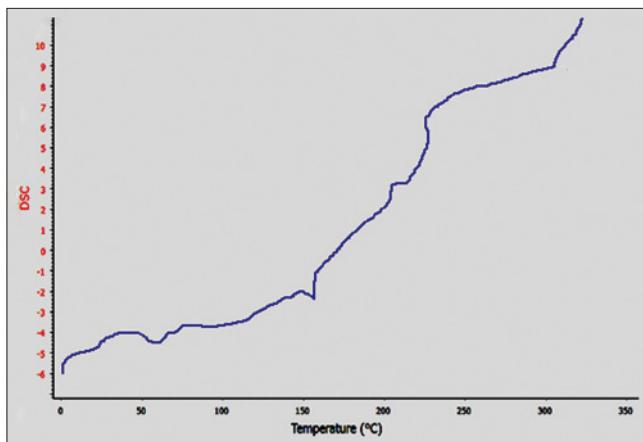


Fig. 7: Differential scanning calorimetry of compound (3)

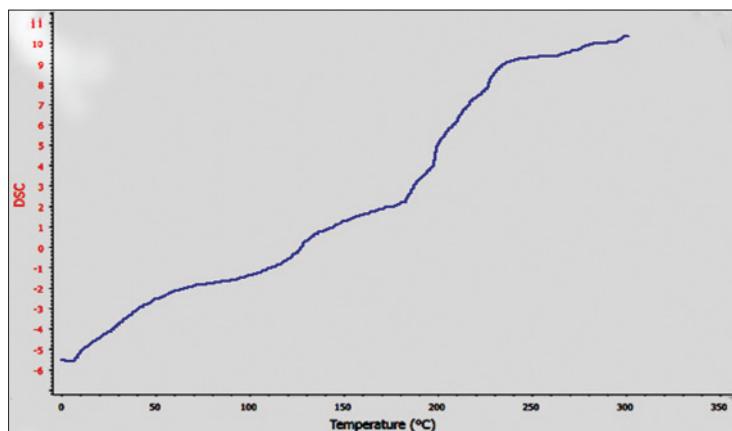


Fig. 8: Differential scanning calorimetry of compound (4)

The results of DSC-measurements gave good evidence of stability of formatted compounds in our work; the formatted compounds appear high resistance toward high temperature due to their components from heteroatoms (sulfur and nitrogen).

Effect of various solvents [14]

The interaction of compounds was tested in series solvents, according to polarity of various solvents, the results are listed in Table 1.

The solvation of prepared compounds depends on solubility and activity of functional group and terminal groups (polarity of group) in solvents which cause interaction act active groups.

Method of antimicrobial assay [18,19]

All synthesized compounds screened with three types of bacteria according to study [18,19]. The bioactivity study was tested at three concentrations (10, 20, and 30) mg/ml concentrations in solvent DMSO using three types of bacteria.

Pseudomonas aeruginosa, *Klebsiella sp.*, and *Staphylococcus aureus* screened at concentrations 10, 20, and 30 mg/ml, respectively. These bacterial strains were incubated for 24 hr at 37°C.

RESULTS AND DISCUSSION

In previously work [1], we synthesized these sulfur cyclic compounds, but now, we will study of antimicrobial activity against three types of bacteria.

Collection of samples and antibacterial assay

According to study [18-20], the biological activity for compounds was tested on four types of bacteria which collected from mouth of patients in the hospital. The antimicrobial results are summarized in Table 2. From results of antibacterial studies, it was found to be potentially activity against all types of bacteria. While antifungal activity at concentrations 10, 20, and 30 mg.ml⁻¹ was summarized in Table 2 and Fig. 11-13.

The three types of mouths, bacteria which tested: *Pseudomonas aeruginosa*, *Klebsiella sp.*, and *Staphylococcus aureus* (Fig. 14-16).

Resistance of bacteria against synthesized compounds

The compounds (1-13) were screened according to their action against bacteria are described Table 2. The presence of monocycles or bicycles bearing nitrogen or sulfur atoms in their structures which increased the biological activity in these formatted compounds.

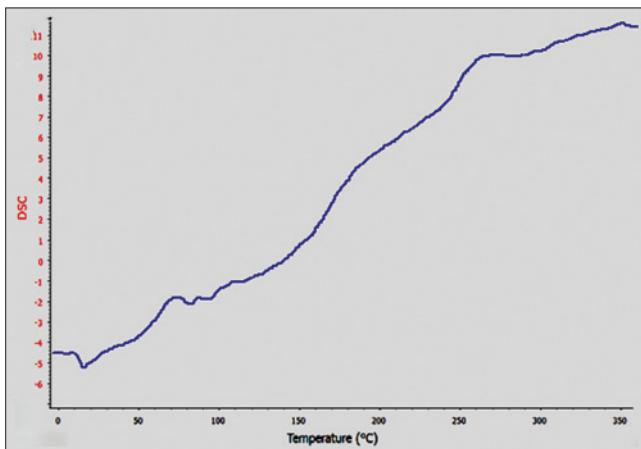


Fig. 9: Differential scanning calorimetry of compound (11)

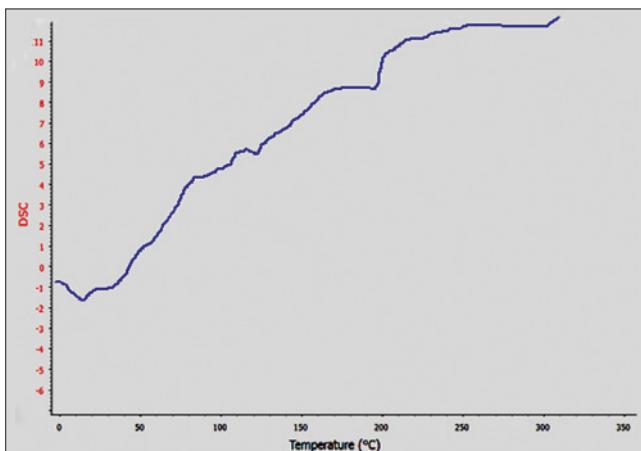


Fig. 10: Differential scanning calorimetry of compound (12)



Fig. 13: Bacteria from Weman sample of burn

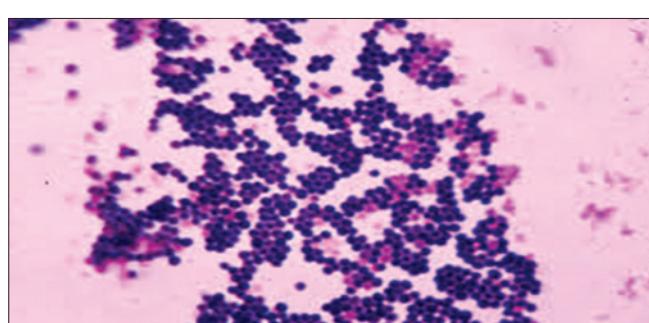
Fig. 14: *Pseudomonas aeruginosa*Fig. 15: *Klebsiella* sp

Fig. 11: Bacteria from Adult sample of burn



Fig. 12: Bacteria from Child sample of burn

The antimicrobial results are summarized in Table 2. From results of antibacterial studies, it was found to be potentially activity against

Fig. 16: *Staphylococcus aureus*

toward three types of bacteria, which gave good data from the results that the biological activity of all compounds has a high biological activity which inhibits the growth of bacteria.

The prepared compounds (11, 13, 12, and 4) have higher activity than other compounds which due to presence of nitrogen and sulfur atoms in their structures [18-20], the mechanism of action for this compounds involved formation of hydrogen bonding with the active centers of the

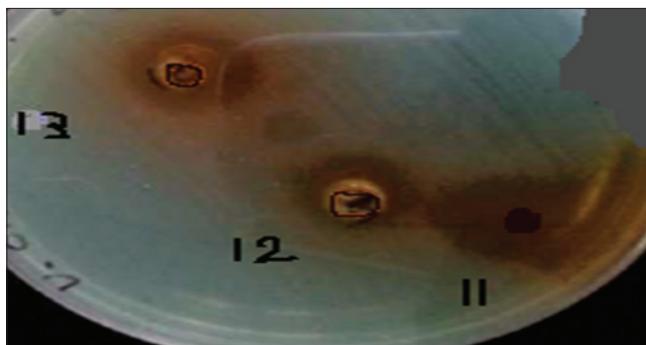
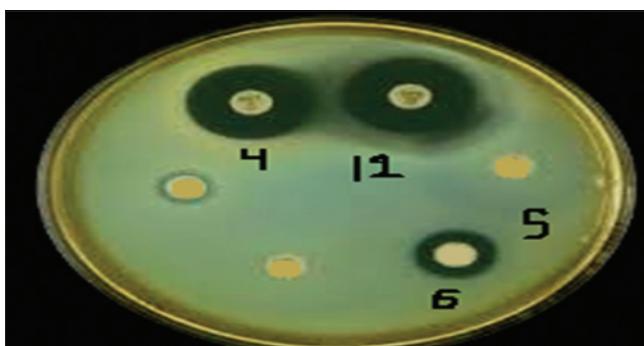
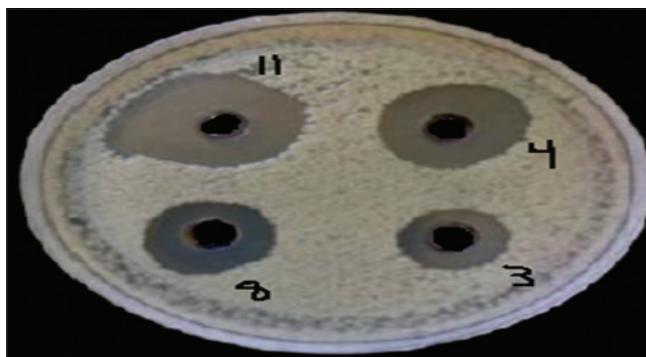
Fig. 17: Antibacterial activity - *Klebsiella sp*Fig. 18: Antibacterial activity - *Pseudomonas aeruginosa*Fig. 19: Antibacterial activity - *Staphylococcus aureus*

Table 1: Solvation of compounds in various solvents

Compounds	Solvents					
	C ₂ H ₅ OH	CH ₃ OH	CHCl ₃	Hexane	CCl ₄	DMF
[1]	+	+	-	-	-	+
[2]	+	+	-	-	-	+
[3]	+	+	-	-	-	+
[4]	+	+	-	-	-	+
[5]	+	+	-	-	-	+
[6]	+	+	-	-	-	+
[7]	+	+	-	-	-	+
[8]	+	+	-	-	-	+
[9]	+	+	-	-	-	+
[10]	+	+	-	-	-	+
[11]	+	+	-	-	-	+
[12]	+	+	-	-	-	+
[13]	+	+	-	-	-	+

cell constituents resulting in the interference with the normal cell process [21,22] (Fig. 17-19).

Table 2: Antibacterial activity of compounds (inhibition zone in [mm]) as average of three concentrations (10, 20, and 30 mg.ml⁻¹)

Compounds	(Average of 3 measurements) <i>Pseudomonas aeruginosa</i>	(Average of 3 measurements) <i>Klebsiella sp.</i>	(Average of 3 measurements) <i>Staphylococcus aureus</i>
[1]	---	6	8
[2]	12	16	18
[3]	10	14	16
[4]	16	16	20
[5]	6	10	12
[6]	10	14	14
[7]	12	16	18
[8]	12	18	18
[9]	---	8	8
[10]	6	10	12
[11]	18	22	22
[12]	16	20	20
[13]	16	18	22

P. aeruginosa: *Pseudomonas aeruginosa*, *S. aureus*: *Staphylococcus aureus*

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