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Article

Preparation of Chalcone-Azo reagents and study of their chemical analysis, thermal studies, and biological effects against fungi

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ABSTRACT

Chalcone reagents are used in various fields because of their biological, medical, and medicinal properties. Therefore, this study aimed to prepare a new reagent of Chalcone–Azo and study its chemical and physical properties using different techniques. In this recent work, four reagents were created and tested using thermal behavior in stability curves, solubility in various solvents, and spectral characterization like UV-visible FT-IR. In addition, a biological study was conducted to estimate the effect of these reagents on two types of fungi. Results showed that spectral characterization methods (UV-visible and FT-IR) showed that new groups of compounds were formed, and starter reactants disappeared. This is good proof of forming new reagents. The outcome of thermal behavior in stability curves solubility in various solvents emphasizes these results. The study of using the new reagents as antifungal substances showed that reagent {3} had the highest effect on inhibiting study fungi growth, followed by reagent {4}.

Keywords: Reagent, analytical, thermal, fungi, bioassay

INTRODUCTION

Chalcone Reagent is an aromatic ketone that forms the central core of various important biological compounds1,2, collectively known as chalcones or chalconoids. Alternate names for chalcone include Benzylideneacetophenone3, phenyl Styryl ketone, benzalacetophenone4, β-phenyl acrylophenone5, γ-oxo- α, γ-diphenyl- α-propylene6 and α-phenyl- β-benzoyl ethylene.7 In a narrow sense, it refers to benzal acetophenone8, but in a broader sense, it is collectively called chalcone9, including its derivatives. Although benzalacetophenone is not found in nature, many chalcones exist.10 For example, there are plant pigments such as biotin (a yellow pigment that contains a glycoside in flowers such as the yellow varieties of dahlia and kibana cosmos)11-13 and Carthamin (a red pigment found

in the crown of safflower, also used as a food coloring in Japan). The closed-loop isomer is flavanone, along with flavonoids14,15 and anthocyanins. These yellow pigment compounds are collectively called flavonoids, a type of polyketide produced16-18 using acetyl CoA as raw materials. Polyketide is a general term for substances that are biosynthesized19,20 via β-polyketide acid represented by the general formula [R (CH2 CO) n CO2 H]. There are two isomers of benzalacetophenone21, cis and trans, but they usually refer to the trans isomer.22-24 It is a pale-yellow crystal with a melting point of 59 °C, but there are many other forms with different melting points25,26 such as 57 °C in ether, 49 °C in carbon dioxide, 48 °C in chloroform27,28, 28 °C in benzene and 18 °C in slightly ethyl alcohol29,30 dimmed by light irradiation (Renji Okazak). As for azo reagent and azo dye compounds31,32, they have a basic principle of introducing two small water-soluble components inside the fibers and then using certain conditions suitable for the occurrence of duplication, giving a larger, insoluble 33,36 and colored molecule. The last component is one of the diazotized primary amines35-37, or the parent amine, when the dye undergoes dialysis. It is used for dyeing cotton materials.38,39 These pigments are characterized by their bright colours40,41 and a high degree of stability.

MATERIALS AND METHODS

Various studies for measurements have been reinforced by maneuvers at the University of Tehran (Iran) and were embodied by Thermo-Curves and Thermal stability in various Temperatures and Fungal-Assay (Tehran University). In Iraq, other analytical studies like solubility, physical properties, infrared measurement, and UV-visible identification.

Formation of Chemical Reagents

Formation of Reagent {1}

The chemical Reagent {1} was prepared by a reaction of 1,4-diaminobenzidine (0.1 moles) in (2 ml) of (HCl), and then a solution of sodium nitrite in the ace path was added. After that, it was added to a basic solution of (0.02 mole) of (4-methoxy benzaldehyde), then naturalized the medium, after that filtrated, washed and dried, to be responsible for new Reagent {1} supportive to procedures.[26,28]

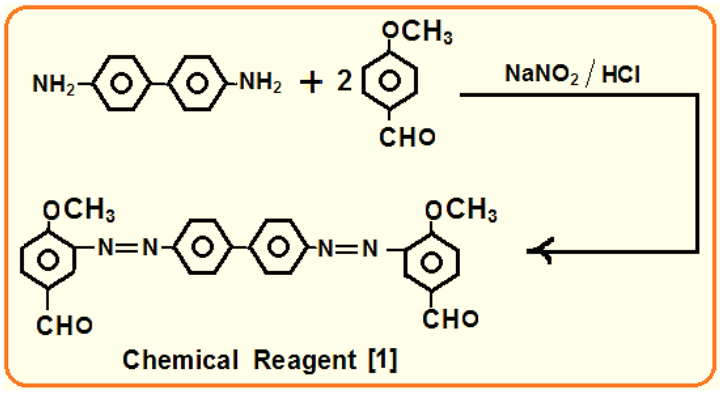

Figure 1: Preparation of Chemical Reagent {1}

Formation of Reagent {2}

The chemical Reagent {2} was prepared by reaction (0.1 mole) of Reagent {1} with 4-chloro acetophenone (0.2 mole) through a rotation step for (7 hours) in the occurrence of (5% NaOH) at room temperature. The next step is embodied by

filtration, aeration, and then a step of recrystallization to afford new reagent {2} supportive to procedures.[26-28]

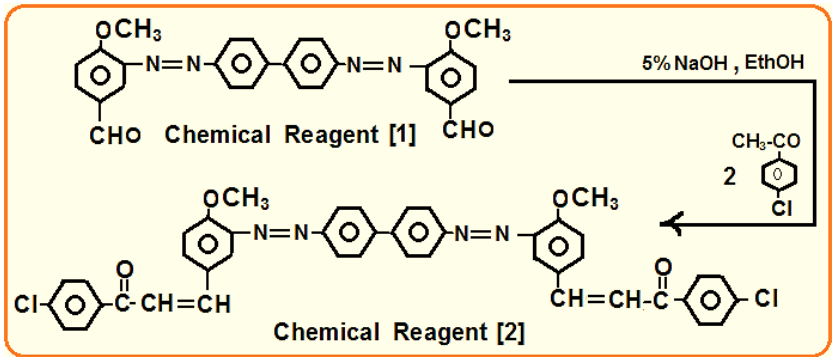

Figure 2: Preparation of Chemical Reagent {2}

Formation of Reagent {3}

The chemical Reagent {3} was prepared by reaction (0.1 moles) of Reagent {1} with 2-aceto thiophene (0.2 moles) through a rotation step for (6 hours) in the occurrence of (5% NaOH) at room temperature, the next step embodied by filtration, aeration, then a step of recrystallization to afford new reagent {3} supportive to procedures.[26-28]

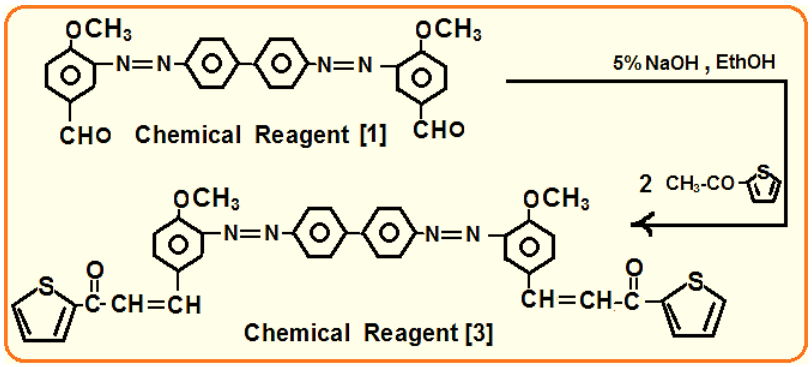

Figure 3: Preparation of Chemical Reagent {3}

Formation of Reagent {4}

The chemical Reagent {4} was prepared by reaction (0.1 moles) of Reagent {1} with 3-acetoindole (0.2 moles) through a rotation step for (9 hours) in the occurrence of (5% NaOH) at room temperature, the next step embodied by filtration, aeration, then a step of recrystallization to afford new reagent {4} supportive to procedures.[26,28]

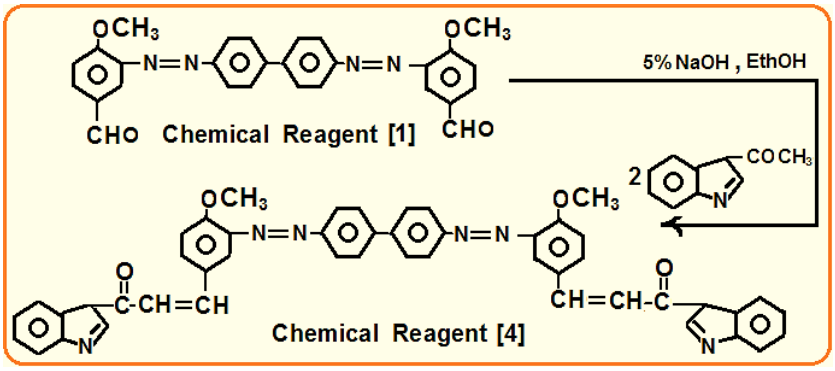

Figure 4: Preparation of Chemical Reagent {4}

Antifungal Assessment[15,16]

The assessment of formatted reagents has been premeditated against some bacteria through the medium (agar) via numerous elegant processes.15,16 The assessment of microbial inhibition was tested at three concentrations: 40, 70, and 80 micrograms with blank solvent (DMSO) per fungi (Aspergillus niger and Candida albicans) with incubation for 24 hours at 37 oC. In this research, different types of fungi were used. These organisms were chosen because of their importance in the medical field, as they cause various diseases and differ in their resistance to antibiotics and therapeutic chemicals. The biological activity was measured by calculating the inhibition area and time, and then the biological activity.

RESULTS

Chalcone reagents have several applications in analytical chemistry, especially in estimating the amount of trace elements through forming metal complexes. Spectroscopic and physical methods can estimate the quantity of these elements. In recent years, chalcone reagents have received great attention as medicinal materials due to the biological efficacy that has appeared in some of them. Fungi are also of industrial importance, as they are used as plasticizers and stabilizers for polymers and gasoline and as initiators of the polymerization process. They are also used as antioxidants and catalysts. For this, the present study involved many studies like spectral studies with thermo-curves and thermal stability in various temperatures and Fungal-Assay:

Spectral Study of Chemical Reagents

UV-Visible –Spectra:

The structure of new Chalcone-Azo reagents proved with Uv-Vis-spectra with perfect concentration in Ethanol as a solvent, all spectral information in figures 1–4:

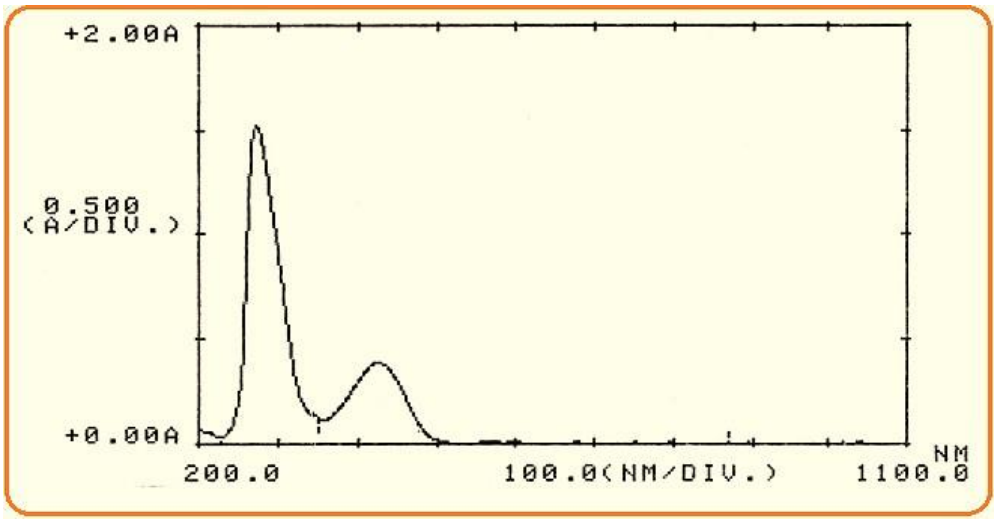

Figure 5: UV-Visible Spectrum of Reagent {1}

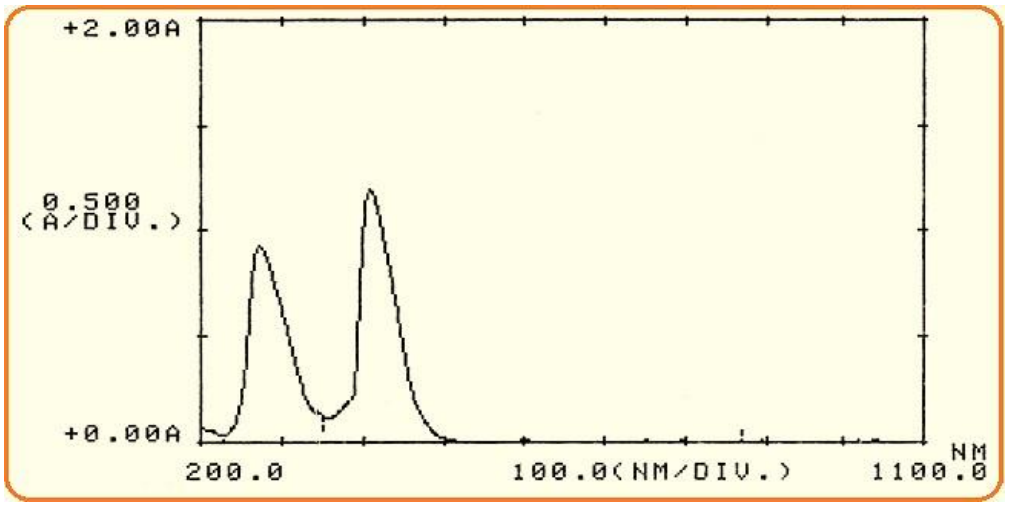

Figure 6: UV-Visible Spectrum of Reagent {2}

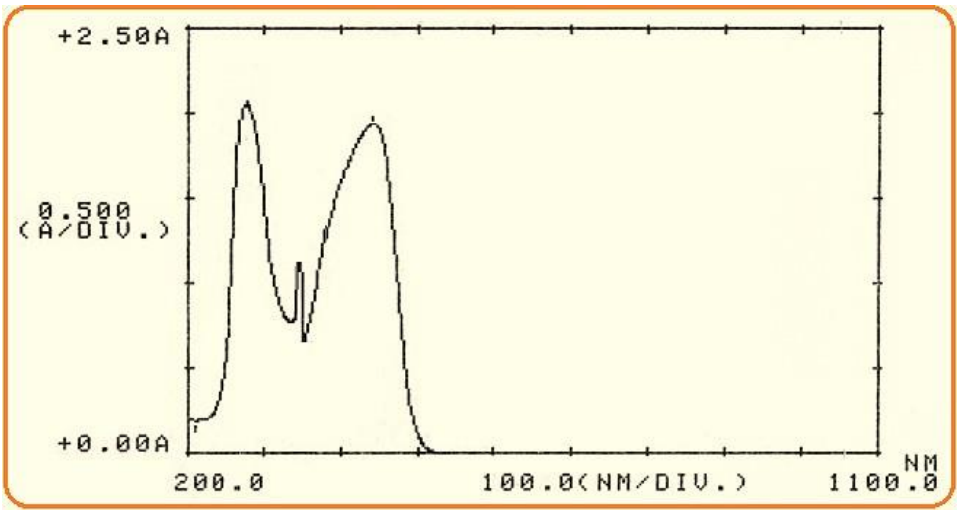

Figure 7: UV-Visible Spectrum of Reagent {3}

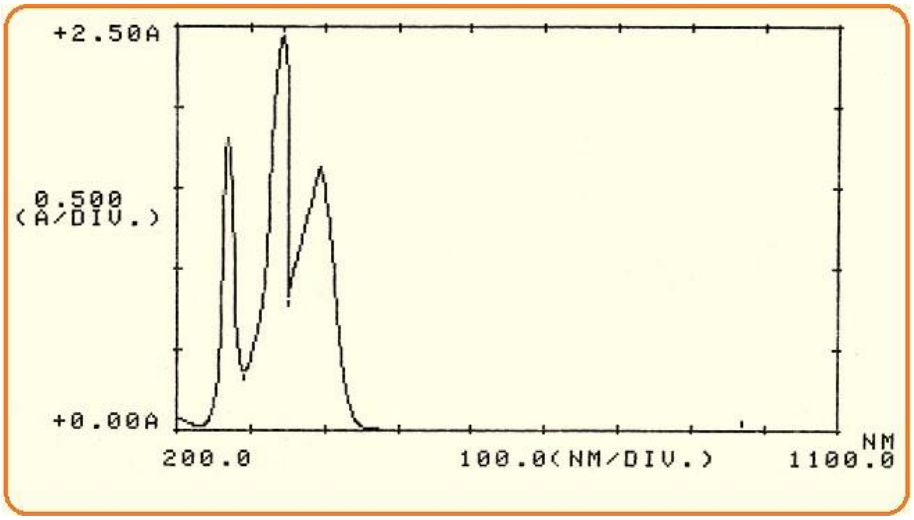

Figure 8: UV-Visible Spectrum of Reagent {4}

3FT-IR- Recognition:

This spectral categorization performed in range (400- 4000) Cm^{-1} to improving our analytical reagents., through this type of measurements, many bands appeared like: bands at [(1472, 1500), (1480, 1504), (1463, 1516), (1488, 1511)] Cm^{-1} for azo groups (-N=N-) in reagents {1, 2, 3, 4} respectively, band at (1706) cm^{-1} for carbonyl group of aldehyde (-CO-H) in reagent {1]., which disappeared and another band appeared at (1689, 1695, 1690) cm^{-1} for carbonyl group of chalcone (-CO-CH=CH-) in reagents {2, 3, 4} respectively, figures (5-8).

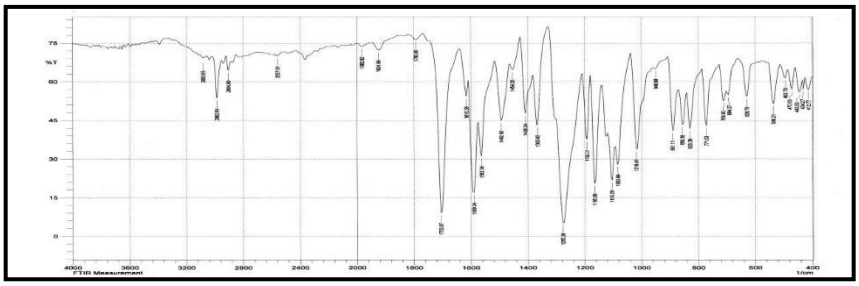

Figure 9: I.R. Spectrum of Reagent {1}

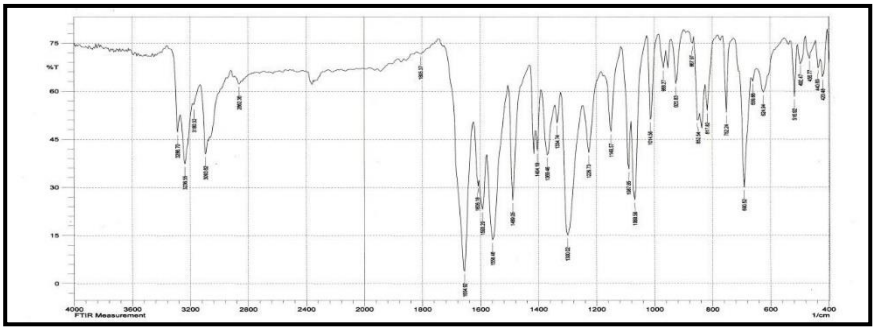

Figure 10: I.R. Spectrum of Reagent {2}

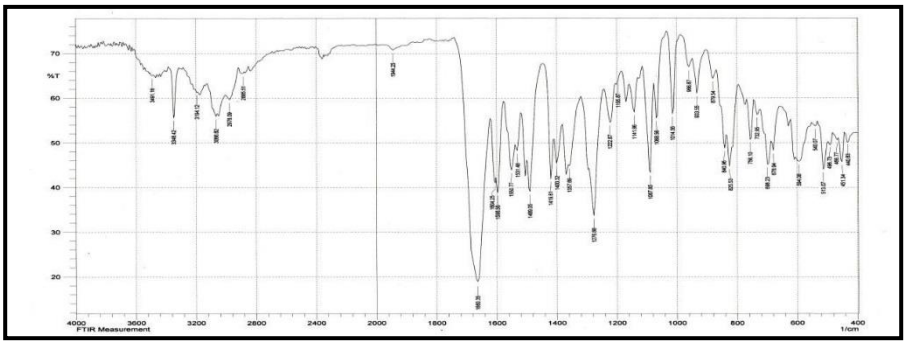

Figure 11: I.R. Spectrum of Reagent {3}

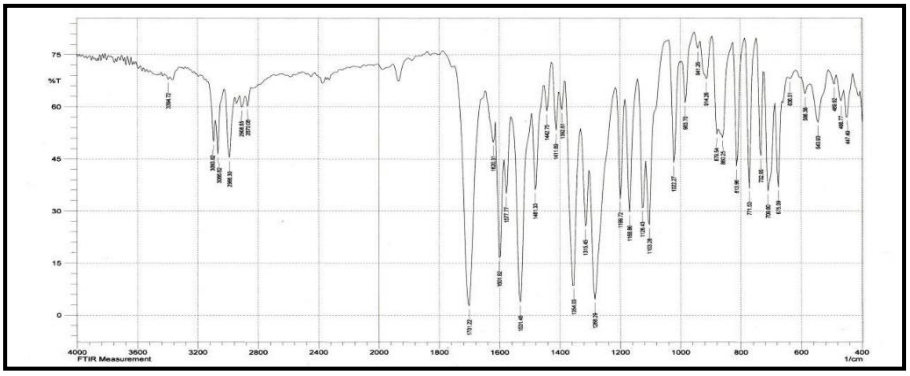

Figure 12: I.R. Spectrum of Reagent {4}

Thermal Studies[8] of Chalcone Reagents

All chambers of Thermo-measurements performed in various temperatures that gave stability of chemical reagents, the reason due to the linkage of Azo group (two groups of Azo -N=N-) with chalcone groups (also two groups of -CO-CH=CH-), figures (9-12):

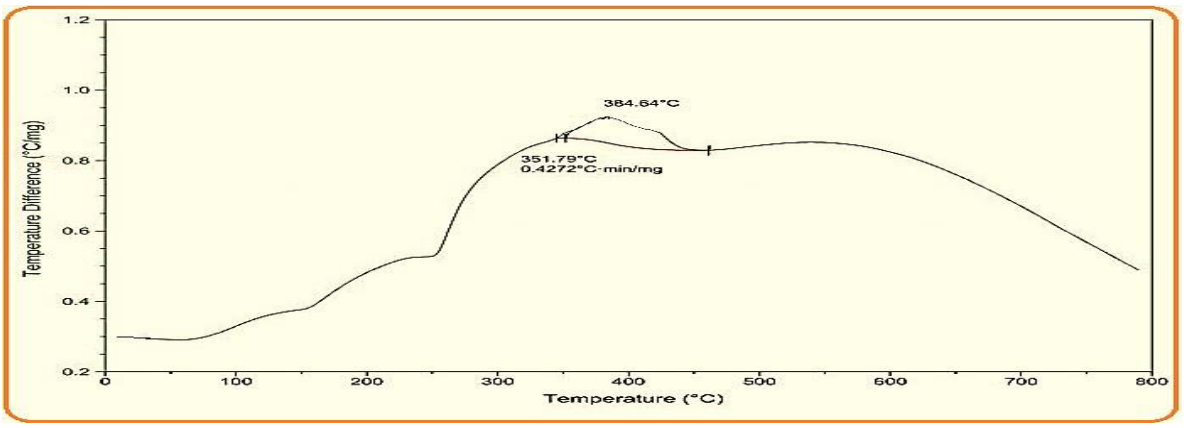

Figure 13: Thermal Curve of Reagent {1}

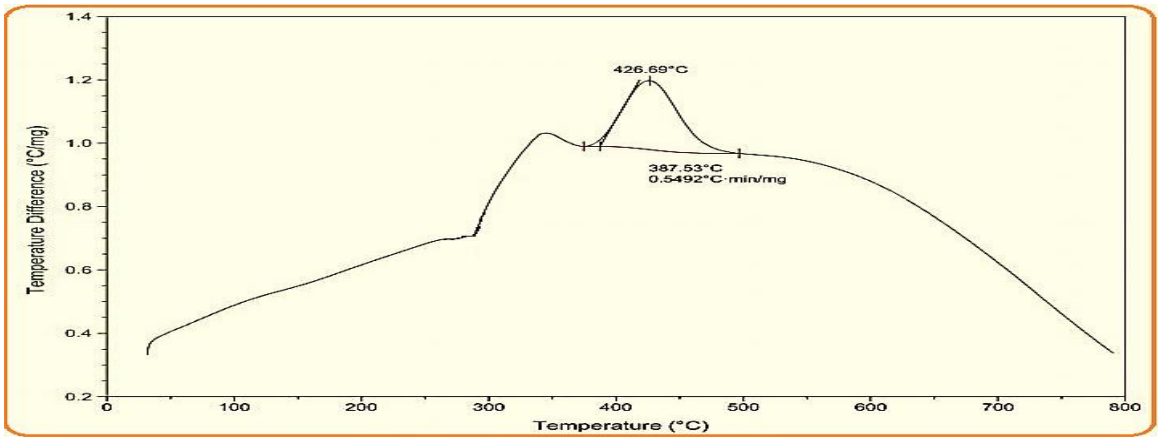

Figure 14: Thermal Curve of Reagent {2}

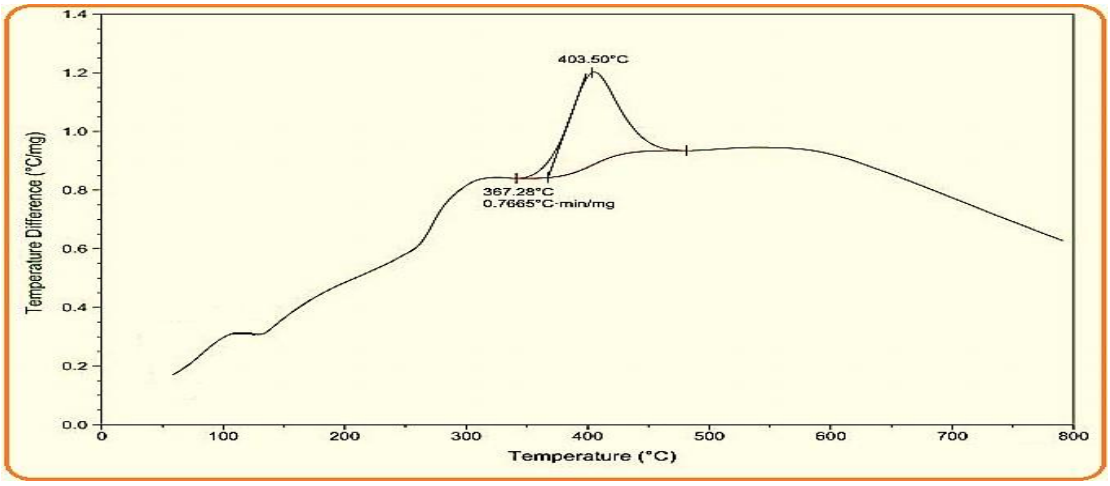

Figure 15: Thermal Curve of Reagent {3}

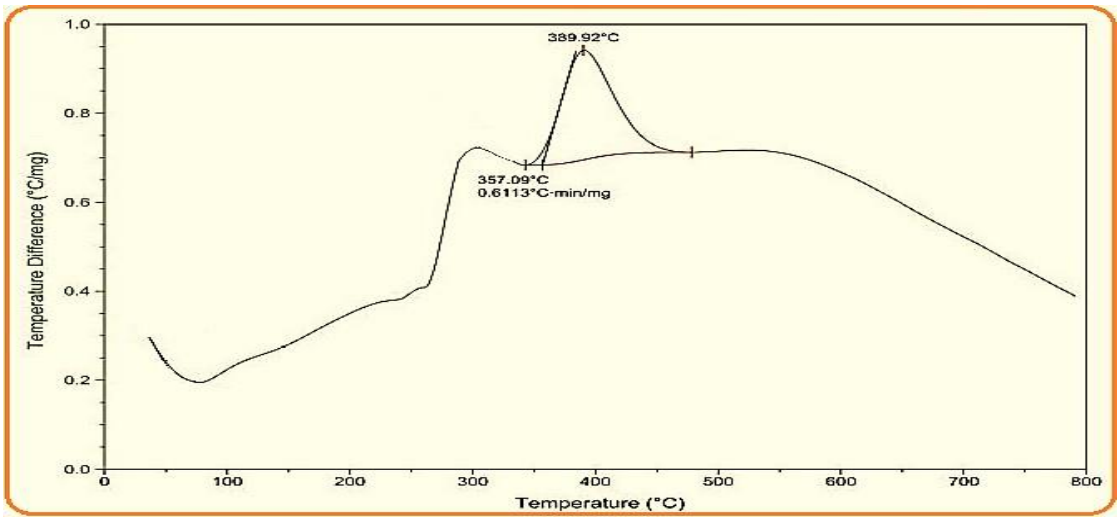

Figure 16: Thermal Curve of Reagent {4}

Behaviour of Reagents in Different Solvents

Conferring to the polarity of formatted reagents, results showed that the reagents have good solubility in some of the solvents due to functional groups in our analytical reagents and structure (nature of active groups) in every compound. The effects are shown in table (1):

Reagents	Meth-anol	Ethanol	DMSO	DMF	Ben-zene	Hexane
Reagent {1}	+	+	+	+	-	-
Reagent {2}	+	+	+	+	-	-
Reagent {3}	+	+	+	+	-	-
Reagent {4}	+	+	+	+	-	-

Table 1: Behaviour of Chemical Reagents in Different Solvents

Physical-Chemical Appreciations:

Physical-Chemical Recognitions and appreciations with data about colors, yields %, in table (2)

Reagents	***Yield %***	***Color***
Reagent {1}	76	Yellow
Reagent {2}	70	Yellowish Orange
Reagent {3}	74	Deep Yellow
Reagent {4}	72	Yellowish Orange

Table 2: Other Chemical-Physical Detections

Antifungal Assessment

Results in table (3) showed that the prepared compounds have different biological activities with different concentrated groups on the base compound, as these new compounds work to eliminate the new pathogenic strains of these types of fungi. The table below shows the results of the effect of the prepared chemicals on fungal growth. Some of the tested compounds generally showed medium and close inhibitory activity against these fungi. The assessment of reagents on kinds of fungi produced vibrant datawith reagents {3 and 4} more than reagents {2 and 1} due to thiophene ring and indole ring in reagents, which increases the inhibition of fungi—results in Table 3.

Reagents	***Candida albicans***	***Aspergillus niger***
Reagent {1}	**+**	**+**
Reagent {2}	**++**	**++**
Reagent {3}	**+++**	**+++**
Reagent {4}	**+++**	**++**

Table.3: Inhibition Test of Reagents in Conclusion (70 micro gram) (+): Inhibition (2-6) mm, (++): Inhibition (8-12) mm, (+++): Inhibition (13-16) mm.

DISCUSSION

The literature supports the total explanation of Spectra in reference. Each reagent gave good stability towards various temperatures, allowing study [8.]

CONCLUSIONS

Numerous guides tested all the formatted new (Chalcone-Azo)- Reagents using investigative and chemical performances and other instrumental methods to ensure that all reagents have good stability in various temperatures according to their structures and good results as inhibitors against fungi.

PATENTS

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Conflicts of Interest: The author declares no conflict of interest.

Ethical Clearance: The Ethics committee confirms that there is no plagiarism and no mistakes or wrong results in this work.

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