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Synthesis characterization of some Schiff bases derived from vanillin containing 1,3,4-thiadiazole moiety

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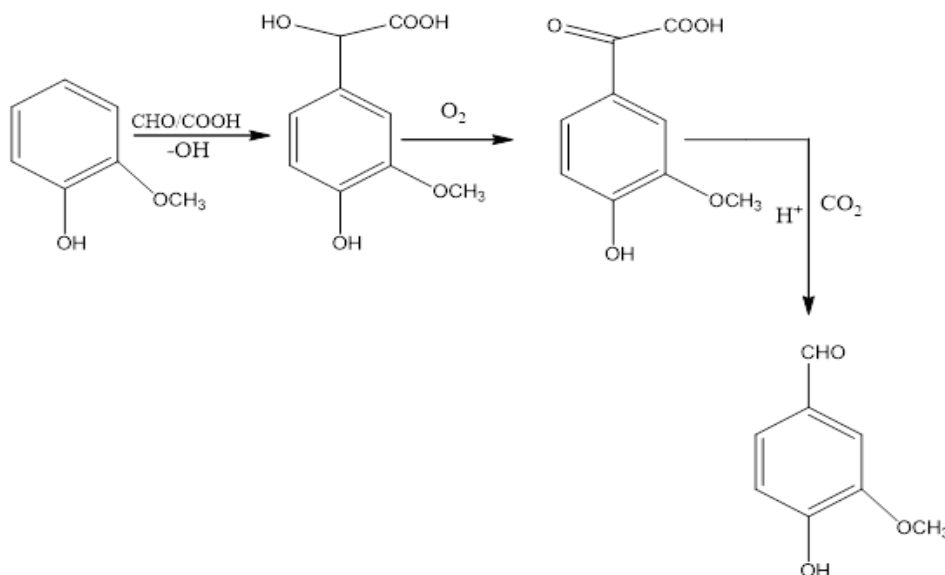
Abstract---The aim of this paper, the Schiff bases 1, 3, 4- Thiadiazol organic Compounds with high pharm. acological efficacy, which play an important role in the treatment of urinary tract infections, as anti-inflammatory, antimicrobial, anti-bacterial, analgesic, pain relief treatment of diabetics, antiviral, and anti-cancer in this research, Vanillin has been used as an important precursor. The Vanillin together, thiosemicarbazide is reacted in the presence of ethanol to yield the substituted thiosemicarbazide compounds which are used in the preparation of several heterogeneous ring compounds include substituents (1,3,4-thiadiazol-2-yl) thiosemicarbazide reacts with Sodium acetate in the presence of a lithium salt and the substituted thiosemicarbazide Compounds are also trapped 4 (5-amino-1; 3; 4-thiadiazol-2-yl) 2-methoxy phenol substituted by its reaction with phosphorus Penta Sulfide- the vehicles prepared were diagnosed by the physical and spectral by IR methods of the nuclear magnetic resonance spectrum (H-NMR)

Keywords---heterocyclic, thiazole, thio semicarbazide.

Introduction

Vanillin is an organic compound (C₈H₈O₃) with a formula and functional groups of aldehyde ether phenol is major component of Vanilla grain extract that crystallizes into white pins, it is found in the pods of the Vanilla plant and is used as an alternative to Vanilla Plant and is used as an alternative to Vanilla material from Vanilla (3-hydroxy-4-methoxy-benzaldehyde) and has other names, Vanilla aldehyde and it is found in Roasted Coffee and Chinese Red Pine, Sometimes Vanilla is used as a synthetic, instead of natural Vanilla extract, as a flavoring agent in Food, beverages and pharmaceuticals, with a distinctive odor and sweet taste, and Vanilla is used as an analytical reagent and intermediate in the Synthesis of preparation Such as methyl dopa antiparkinsonian, in addition

to sickle cell anaemia, it is an anti Fungal and is used to cover the odour of certain combinations such as Caffeine and Polythazide diuretics and antihypertensive tablest it is native to mexice, but now grows. in the tropics, Madagas car is now the largest Producer of vanillan (B-D-gly coside), has no taste odor after harvest⁽¹⁾, Adistin cive Flavor develops after the processing process. and lasts a whole moth, But-in general, it is as follows:



Vanilla is used in skin care products such as skin, Skin and lip moisturizers an acne-a cne therapy Vanilla ahelps to purge the skin and reduce the appearance of pengetbaskip and food blisters Vanilla also Containse a good source of Vitamin (B6) an vitamin (B12) and pan the nic acid it also helps to slow the onstet of signs of aging such as wrings and is used in Comsmetics for Fragrance⁽²⁾. Also to treat burns and wounds, Vanilla extract, which is focused directly on open. burns and wounds, should not be applied to hair when blending Vanilla with other ingredients such as almond oil and castor oil and applying for 15 minutes to hair that makes it soft, single and Shining, Cough, nausea, and depressio depression⁽³⁾

Experimental

The melting pont of the prepared vehicles and all of my company's materials was measured (Fluka) and (BDH), the infrared spectrum of some models Was measured using (Frans form FTIR -84005) and the (H-NMR) has been measured for a number of the prepared using (HNVMR), Bruker Analyische messte chink GmbH j400 MHZ). and the remote has been use chem Bio 3D ultrall the digital scale, a sublimatory a heater, and a hydrant were, to pull out gases and Vapors .

Synthesis of 4-substituted 2-(4-hydroxy-3-methoxy benzylidene) thiosemicarbazide

ThiosemiCarbuzd (0.1 mi) and Crystalline & Sodium acetate (0.03 M) were taken RB Flask, (8-10Ml) water an (0.3gm) of aldehyde was added. Slowly continuous stirring added The mixture, was turbid so added methanal abs. until clear. Solution obtained shake Mixture For few minutes and allowed to stand - thiosemicarbazone precipitated from the Cold Solution-filler off the precipitate and recrystallize with ethandiaabs⁽⁴⁾. other compounds were prepared Similary and their character, action date are recorder in Table (1)

Table(1) Some physical constant for thosemicar bacide Compounds (E₁, E₂)

Comp.No	R	Molecular Formale	M.P (°C)	Yield (%)	Color
E ₁	Ph	C ₁₂ H ₁₂ N ₅ O ₄ S	148	83	White
E ₂	CH ₂ CH=CH ₂	C ₁₇ H ₁₈ N ₅ O ₂ S	195	82	White

Synthesis of 4-(5-amino-1,3, 4-thiadiazol -2-yl) 2-methoxy Phenol

Thios micarbazone (0.1M) and acetate Sodium(0.03m) were dissolved in (20-30ml) of glacial acetic acid taken inevoud-bottom Flask aquipped with a separating funnel for the addition of bromide. Bromine (0.6ml in 4ml glacial acid) was added slowly to it, white stirring magnetically After half an hour Stirring, the solution was Poured on crushed ice, the resulting Solid was separated, dried recrystallized from⁽⁵⁻⁶⁾. Ethanol .abs, other compounds were prepared Similary and their characterization date are recorded in Table(2)

Table(2) Some Physical Constant for Compounds (E₃, E₄)

Comp.No	R	Molecular Formale	M.P (°C)	Yield (%)	Color
E ₃	CH ₂ =CH- CH ₂	C ₁₂ H ₁₃ N ₅ O ₂ S	148	54	Dark yellow
E ₄	Ph	C ₁₇ H ₁₉ N ₅ O ₂ S	195	82	White green

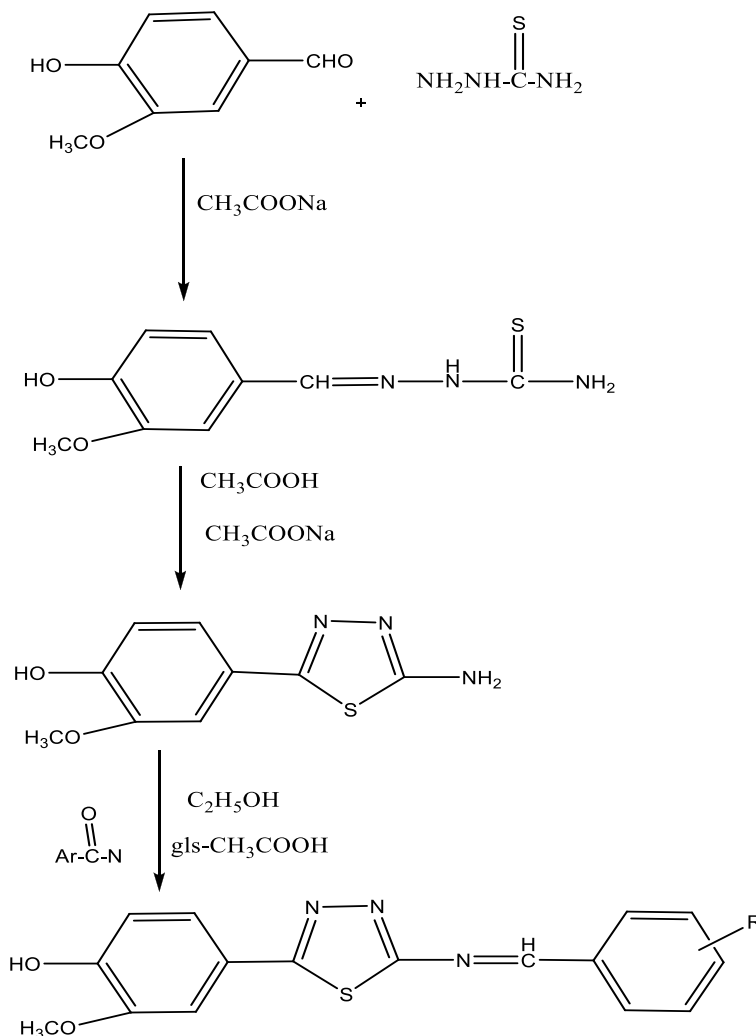
Synthesis of Sheff bass 2-methoxy-4-(5-(4- methyl benzylidene amino) - 1,3,4-thiadiazol-2-yl) phenol

Asolution of (0.1M) was prepared in 15ml Alcohol. bottom Flask Required aldehyde, (0.1m) dissolved in (10Ml) alcohol, was then added to it. the mixture was refluxed for (6-7hr). the volume of alcohol, for wase reduced to half by distillation under reduced Pressare the resulting solution was poured on Crusheddice the precipitate which get separated was dried and recrystallized From ethanolabs⁽⁶⁻⁷⁾, Other Compouds were prepared similary and their characterizationdate are recorde in Table (3)

Table (3) Some physical Constant for compounds (E₅, E₆)

Comp.No	R	Molecular Formale	Yield (%)	M.P (°C)	Color
E ₅	Ph	C ₁₇ H ₁₉ NOSCL	75	175	White
E ₆	Ph	C ₁₇ H ₁₉ N ₅ O ₂ S	195	82	White green

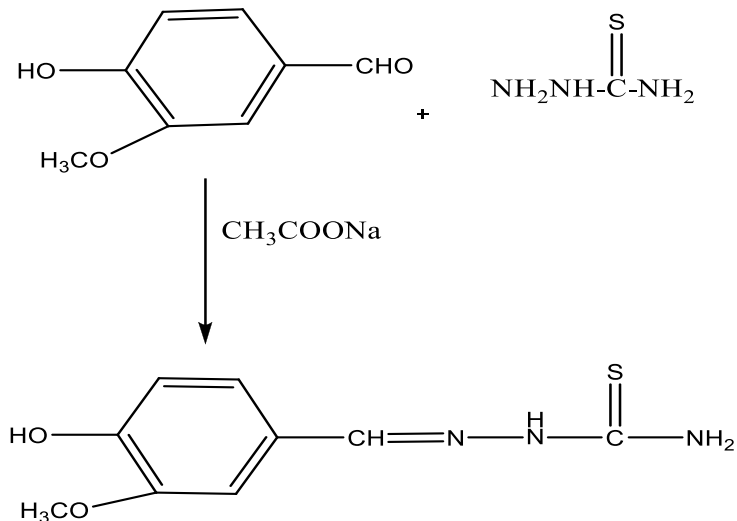
Result and Discussion



Marly studies have shown that heterogeneous ving compounds are important in medicine as therapeutic sachst Substances, industrial dyes, and antibiotics and in Stimulants of in agriculture as triggers and growth

Synthesis of thiosemicarbazone (e₁)⁽⁸⁻⁹⁾ .

Thiosemicarbazone compounds are important because they can be converted to diazole derivatives when reacted with different reagents derived from the reaction of vanillin in the presence of ethanol abs. as shown by the following equation:-



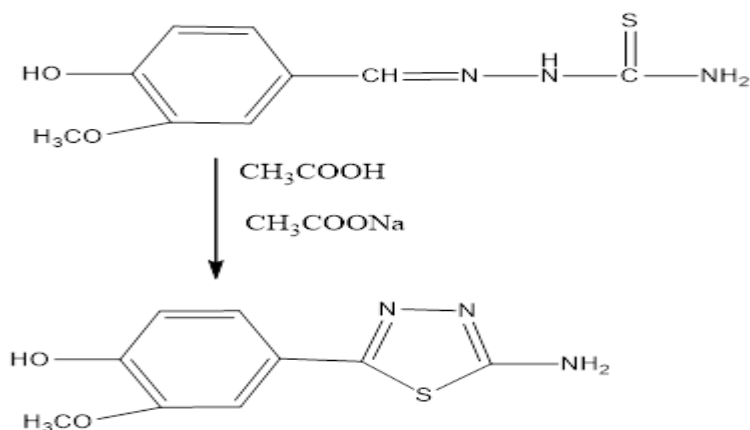
The infrared spectrum of the compound shows frequency bands at 3290 cm⁻¹ (strong C-H stretch), 1157 cm⁻¹ (C-C stretch), 1249 cm⁻¹ (C=N stretch), 3070 cm⁻¹ (C=S stretch), and 1600 cm⁻¹ (N-H stretch). The ¹H NMR spectrum (CDCl₃) shows peaks at 6.7-7.8 ppm (4H, Ar-H), 3.8 ppm (t, 3H, OCH₃), 5.8 ppm (s, 1H, OH), 8.2 ppm (t, 3H, CH₃), and 2.0 ppm (t, 2H, NH₂)⁽¹⁰⁻¹²⁾.

Table (4) Spectral data for synthesis compounds (e₁-e₂)

Comp.No	R	N-H	C-H	C-C	C=N	C=S
e ₁	Ph	3090	3290	1157	1391	1249
e ₂	CH ₂ CH=CH ₂	3217	3100	1147	1595	1266

Synthesis of 4-(5-amino-1,3,4-thiadiazol-2-yl) 2-methoxy Phenol

Reaction of thiosemicarbazone with sodium acetate in ethanol abs:-



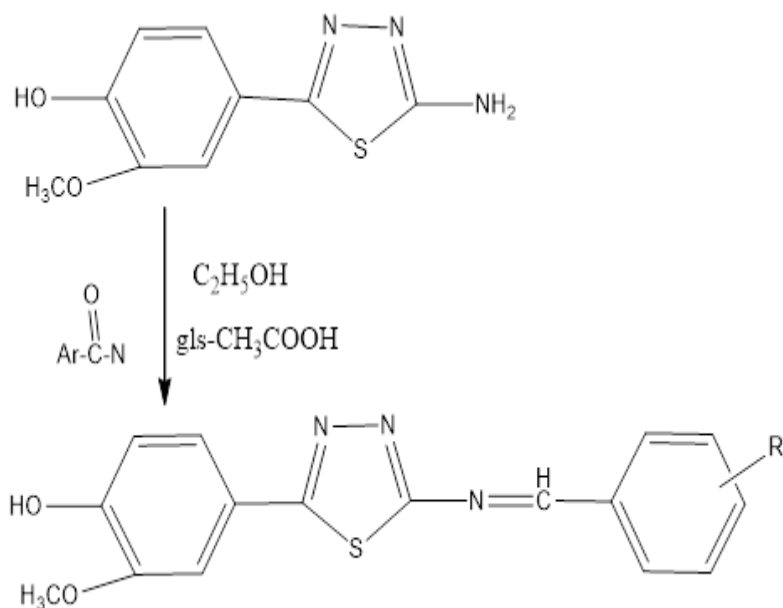
The Infrared spectral Copy the (IR) (3385cm^{-1}) (C-H); (1157cm^{-1}) (C-C), (1691cm^{-1}) (C=N), (1567cm^{-1}) (N=C), (949cm^{-1}) (C-S), (3120cm^{-1}) (OH), (1096cm^{-1}) (C-O), (1674cm^{-1}) (C=N), ¹H NMR (CDCl_3), 8 (6.7), (H, Ar-H), 8.8-7.3 (M, 4H, $\text{C}_6\text{H}_5\text{CH}=\text{N}$) 8.4 (S, 1H, OH), 8.8 (t, 3H, $\text{C}_6\text{H}_5\text{CH}=\text{N}$), 3.3 (t, 3H, OCH_3)

Table (5) synthesis of 4-(5-amino-1,3,4-thiadiazol-2-yl)-2-methoxyphenol

Comp.No	R	O-H	C-H	C-C	C=N	N=C	C=S
e ₃	Ph	3385	3387	1157	1691	1674	949
e ₄	$\text{CH}_2\text{-CH}=\text{CH}_2$	3290	3290	1246	1646	1670	930

Synthesis of Schiff bases of 2-methoxy-4-(5-(4-methoxy-4-(5-(4-methylbenzylideneamino)-1,3,4-thiadiazol-2-yl)phenol

The reaction Alcohol, Required and aldehyde and then added to it th Mixture:-



The newly Synthesis Compound of Schiff bases were characterized by Using IR and HNMR spectroscopy, the Compounds showed Peaks at (3290-3271)(N-H) stretching (2935-2932 cm^{-1}) (C-H) Stretching (1170-1157) (C=N) stretching, (1631-1624), C=S Stretching / (1249-1216) cm^{-1} the NMR Spectrum of the Compound Showed, (6-7) (M,4H, Ar-H), (6-8-7.4) (M), ($\text{C}_6\text{H}_5\text{CH}=\text{N}$), (5-0 S,1H,OH) S8(S,H,C₆ H₅ CH=N)⁽¹³⁾.

Table (6) indicated the presence of aromatic proton

Comp.No	R	N-H	C-H	C-C	C=N	C=S
e ₅	Ph	3290	2935	1170	1691	1249
e ₆	CH ₂ -CH=CH ₂	3271	2932	1147	1624	1216

Conclusion

Atatal 6 compounds were synthesis with good gields All synthesis Compounds is exhibited analgesic anti-inflammatory and Antibacterial activity, The Compound (e₁,e₂,e₃,e₄,e₅,e₆) were shown significant analgesic activity against swiss albino. Vats were compounds

References

1. Carrillo, D. E. A., Flores, N. O. B., Roman, J. F. J., Medranda, M. B. B., & Alvarado, C. A. H. (2022). Photovoltaic system to improve energy efficiency. *International Journal of Physical Sciences and Engineering*, 6(1), 18–26. <https://doi.org/10.53730/ijpse.v6n1.3146>
2. D. N. Satyan arayana, electronic Absorption spectroscopy and related technique university press india limited new delhi. (2001)
3. Hassan, A.S. And Hafaz. T.S. Antimicrobial activites of ferrocenyl complexes A review J. App. pharm. Sci,(2018), 156-165.
4. Hogarth, G. metal-dithicarbamate complexes , chemistry and biological activity minireo-med , chem, (2012), 12.
5. Ismailova, Z., Choriev, R., Khimmataliev, D., Mustafoeva, D., Hashimova, M., Ochilova, G., Fayzullaev, R., & Berdalieva, G. (2022). The forensic professional education teacher mediacompetery development technology. *International Journal of Health Sciences*, 6(2), 1189–1205. <https://doi.org/10.53730/ijhs.v6n2.11785>
6. Kol, Parks, lee g,kim h,an efficient one- pot synthesis of 2,5- disubstiuted 1,3,4- thiadiazoles from aldehyds andhydrazides using lawessonnes reagent , arkiroc, (2019), 67-68.
7. Kuar S.P.Rao R. and Nanda S.Amoxicillin, Abroad spectrum antibiotic, Interntional Journal of pharmacy and pharmaceutical sciences, (2011), (3)
8. S. H.Saroj , R.S and dwiredi D. operationally simple green synthesis of some Schiff bases using grinding chemistry technique and evaluation of antimicrobial acitivites green process synth, (2012), 464-477
9. S.A.K. B and Raja. J. D. Recent advance in metal complexes derived and their potential , J.Mol . struct (2020) 1222
10. S.M .A. M. Isloor, D.S, Akhila, H. Fun, synthesis characterization and antibacterial activity of some new pyrazole based Schiff bases , arab J.chem ,(2013),334-340.

11. Shah V H, Vashi B S, Mehta D S Indian journal of chemistry (1996), 35, 11-115.
12. Shel drick, G.M. Crystal structure refinement with shelxl acta cryst, (2015), 27.
13. Shu. H, Yu, N. Sixie, G.L, Chen aniline, methyl phenol, Acta cryst, (2011).67.
14. Svejstrup, T.D, Ruffoni, Julia , F, Aubert, V.M, Leonori, D. synthesis of Aryl amine via aminium radicals, angew.chem (2017),129.
15. Widana, I.K., Sumetri, N.W., Sutapa, I.K., Suryasa, W. (2021). Anthropometric measures for better cardiovascular and musculoskeletal health. *Computer Applications in Engineering Education*, 29(3), 550–561. <https://doi.org/10.1002/cae.22202>
16. Z. M. Abdallah S. synthesis and structure in vestigation of the antibiotic complexes of d. blok elements spectrochimica Acta part A, Moleclar and Biomolecullar spectroscopy (2005), 61(9)