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"Studies On Properties, Synthesis And Characterization Of Substituted Heterocyclic Compounds – A Review"

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Abstract :

Our study deals with the anti-microbial activities, synthesis, properties and spectroscopic characterization of five membered ring containing heterocyclic compounds. Naphthol and their substituted derivatives are the novel series of cyclic compounds which contains sulphur and nitrogen heteroatoms. These compounds shows anti-bacterial as well as anti-fungal activities compound are synthesized by various methods. The structures of heterocyclic compounds are described by FT-IR, ^{13}C-NMR, ^{1}H-NMR etc, techniques. These derived heterocyclic compounds shows anti-bacterial activities against various Gram positive as well as Gram-negative bacteria, whereas, anti-fungal activities against the Candida species, Aspergillus species and Albigo species.

The synthesized compounds shows greater anti-microbial activity than the noval compounds. The main aim of our research is deals with the substituted heterocyclic compounds shows potent biological activities.

Keywords : Heterocyclic compounds, Naphthaol, Substituted Naphthol, anti-bacterial activity, anti-fungal activity, substituted reaction, Thiazole etc.

Introduction :

Oxygen, sulphur and nitrogen atom containing heterocyclic compounds are the most common and these compounds are very important type of organic compounds because their applications in various sectors like industrial, pharmaceutical, medicinal, drug design, agriculture etc, shows by several researchers. Three compound reactions in chemistry is very important due to their diversity and brevity.

Thiazoles are nitrogen and sulphur atoms containing heterocyclic compounds explained by Toche and Deshmukh in 2017, which shows several biological activities, proofed by Ayati et al. in 2015, Rouf and Tanyeli in 2015 etc.

Thiazoledin-4-ones are the stable form of thiazole with ketones, according to Kumar and Patil in 2017.

Thiazolidinones are important heterocylic compounds because their ring system is the core structure of the variety of derived synthetic compounds, according to Abdullah in 2014. These compound shows potent biological activities shown by Kapoor et al. in 2016 and Nirwan et al in 2019.

Unsaturated five membered heterocyclic compounds like 1,2-dihydro-3H-1,2-4-triazol-3-ones containing three nitrogen atoms shows potent biological activities explained by Shneine and Alaraji in 2016 and Kaur and Chawla in 2017. Due to potent anti-microbial activities five membered heterocyclic compounds with two hetero atoms, synthesis is very important in the medicinal point of view, and study of their derived compounds are also important.

Experimental :

A. Instruments :

(i) Scientific Capillary melting point. It determines melting points of the compounds.

(ii) Thin layer chromatography (TLC) technique to determines completeness of the reactions on pre-coated silica gel aluminum plates.

(iii) n-hexane : methanol chloroform in the ratio of 5:2:3 or used as an fluent.

(iv) FT-IR spectroscopy were recorded.

(v) ^{1}H-NMR and ^{13}C-NMR spectra recorded, using TMS as internal standard and DMSO as a solvent.

B. Methods :

(i) Synthesis of 1-thiocarbamido alkyl-2naphthol syntheiszed by Nagawade and Shinde in 2007 and Younis et al in 2012.

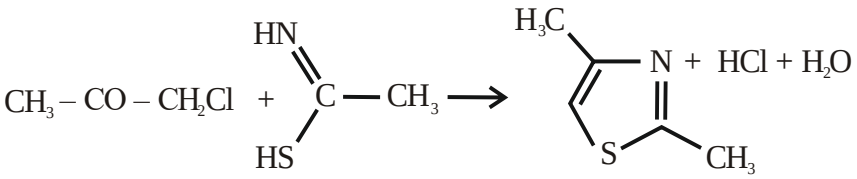
Round bottom flask contains mixture of 2-naphthol (1.44g,0.0/mol), substituted benzaldehyde (1.02 ml) benzaldehyde (1.51 g), 3-nitrobenzaldehyde and 4-nitro benzaldehyde (0.01 mol), $ZrOCl_2.8H_2O$ (0.032 g, 0.1 mol) as a Catalyst, thiourea (0.91g, 0.012 mol), in 1,2-dichloromethane (15 ml) as a solvent were irradiated in an ultrasonic cleaner bath at room temperature for 12-20 minutes, monitored by TLC, then reaction mixture was cooled and then 20 ml H_2O was added and stirred, then precipitation starts, precipitate was filtered off and recrystallisation from ethanol.

(ii) Synthesis of 2-(2-hydroxynaphthalen-1-yl) (4-nitrophenyl) methylhydrazie-1-carboxamide, synthesized by Pouramiri and Kermani in 2017.

A solution of 2-naphthol (1.44 g, 0.01 mol), semicarbazide (1.226g, 0.01 mol), 4-nitrobenzaldehyde (1.51 g, 0.01 mol) and indium (III) chloride (0.022g, 0.1 mol) in absolute ethanol (15 ml) with chloroacetic acid (1.89 g, 0.02 mol) were irradiated in ultrasonic bath at room temperature. The reaction was monitored by TLC, after the filtration, washing and recrystallisation from ethanol compounds was obtained.

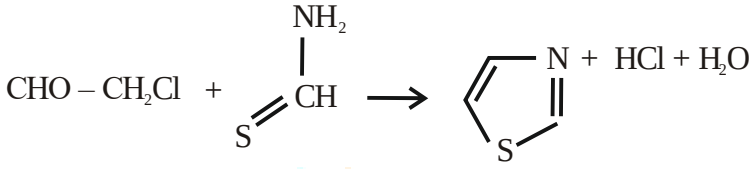
(iii) Synthesis of thiazoles :

❖ by the interaction of chloroacetone and thioamides



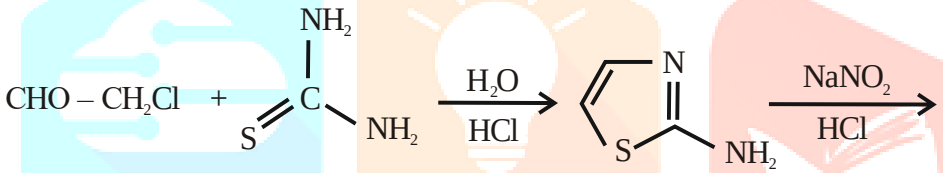
α – thioacetamide thioacetamide 2,4-dimethylthiazole

❖ by the interaction of chloroacetaldehyde and thioformamide



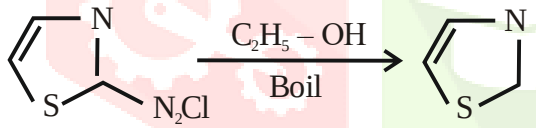
α - chlroacetaldehyde thioformamide thiazole

❖ by the interaction of chloroactaldehyde and thiourea



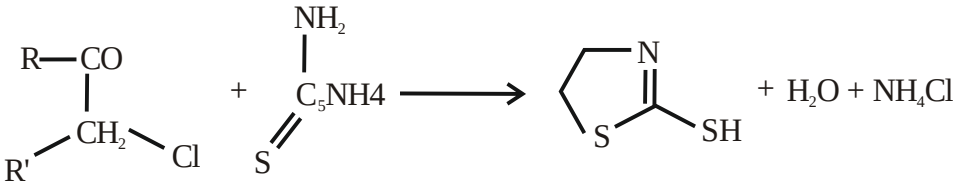
α - chlroacetaldehyde thiourea 2-aminothiazole

❖ by the interaction of α-halocarbonyl compounds and ammonium dithiocarbamate.



thiazolediazonium Chloride thiazole

❖ **By interaction of α-halocarbonyl compound and ammonium dithiocarbonate**



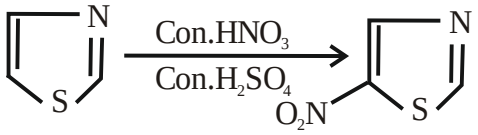
Characteristic of Thiazole :

(1) It is less reactive than imidazole but in intermediate between pyridine and thiophene.

(2) Electrophilic substitution takes place at position-5.

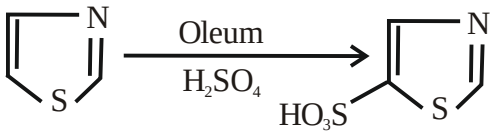
(3) In acidic medium, the attack takes place through the formation of thiazolium cation.

(4) Nitration reaction



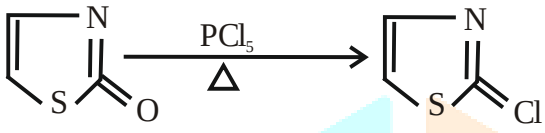
Thiazole **5-nitrothiazole**

(5) Sulphonation reaction

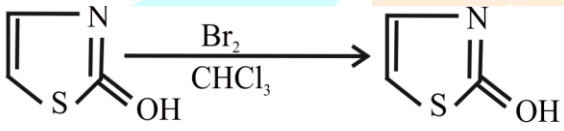


Thiazole **Thiazole-5-sulphonic acid**

(6) Halogeneation reaction



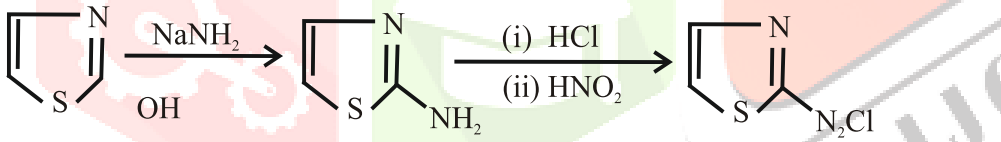
2-thiazolone **2-chlorothiazole**



2-hydroxythiazole **5-bromo-2-hydroxythiazole**

(7) Hydrogenation reaction

(8) Diazotisation reaction



thiazole **2-aminothiazole** **thiazolyldiazonium chloride**

(9) Oxidation reaction : Thiazole ring is resistant to oxidation by nitric acid but the ring is cleaved by potassium permanganate.

(10) Reduction reaction : Thiazole ring is resistant to the reduction reaction by many reducing agents, but raney nickel can be used for desulfurization reactions.

(11) Troglitazone, Rosiglitazone, Pioglitazone, famotidine, sulfathiazole, phthalylsulphathiazole, meloxicam, thiabendazole etc, are derived from thiazole compounds is used as potent medicines, which cure different diseases.

(iv) Synthesis of 2-(2-((2-hydroxynaphthalen-1yl) (phenyl) (methyl imino)-4- oxothiazolidin-5-yl) acetic acid by Sushil Kumar and Devanand in 2003.

Taken 100 ml round bottom flask fitted with reflux condenser, compound ((2-hydroxynaphthalen-1-yl) (phenyl) (methyl) thiourea (0.616g, 0.002 mol) and maleic anhydride (0.196 g, 0.002 mol) in glacial acitic acid (20 ml) were refluxed with stirring for 10-12 h, reaction monitored by T.L.C, the reaction mixture reduced to half and cooled at room temperature, after filtration, dried and crystallisation from ethanol compound was recovered.

(v) Synthesis of 1-((2-hydroxynaphthalen-1-yl) (4-nitrophenyl) (methyl)-5-phenyl-1, 2-dihyro-3H-1,2,4-triazole-3-one, by Shalini et al in 2009.

Benzoyl chloride (0.28g, 0.002 mol) and compound 2-((2-hydroxynaphthalen- 1-yl) (4-nitrophenyl) (methyl) hydrazine-1-carboxamide (0.704g, 0.002 mol) were dissolved in ethanol, potassium carbonate (0.42g, 0.003 mol) added, the mixture was refluxed for 6-8 hours, then heated in alkaline medium 4% NaOH (20 ml) for around 4-6 hours, the reaction mixture was neutralized by dilute HCl after this solvent was evaporated and product was recrystallized from ethanol.

- Anti-bacterial activity, shown by Landage et al 2019. Synthesized compounds shows anti-bacterial activity determined in vitro against Gram positive strains and Gram negative strains by agar well diffusion method.

- Anti-fungal activity, shown by Pejchal et al. in 2015. Synthesized compounds shows anti-fungal activity mostly the Candida species in Muller Hinton agar medium.

Results and Discussion :

In this study heterocyclic compounds and their derived compounds like thiazole, 4-thiozolidinone etc. were synthesised from the various methods (discussed earlier). The FT-IR spectra of the starting materials are changed, because cyclization in synthesized compounds occurs. NH_{2str} peaks disappears, whereas C = $N_{str.}$ peaks are appeared in synthesized compounds at 1600-1766 cm^{-1} shown by de Aquino et al. in 2008.

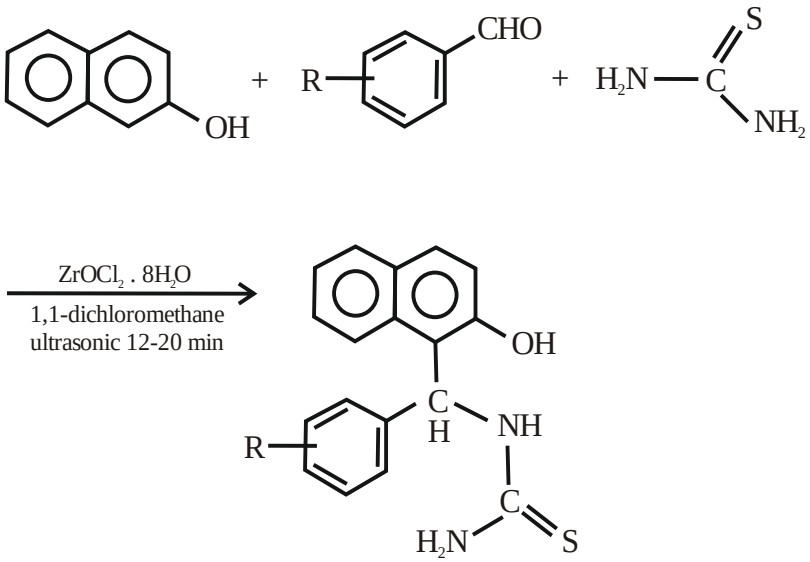
^{1}H-NMR spectra of the compounds are changed after cyclization. The protons of NH_2 groups are disappears in cyclic synthesiszed product, whereas these two protons are present in several compounds at 8.16, 9.18, 9.13, 6.71 ppm. Singlet band of $CH_{thiazole}$ are appeared shown by Bhosale et al. in 2012, a singlet band of OH, doublet bands of CH_2 and triplet bands of $CH_{thiazoledin\text{-}4\text{-}one}$ are appears, shown by Gurumurthi et al. in 2009.

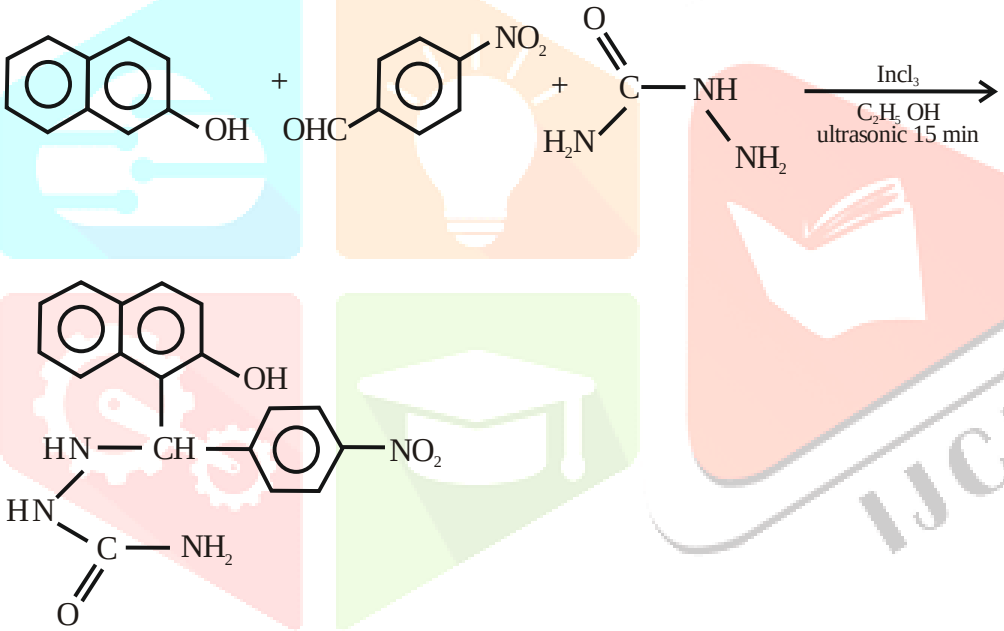
Two protons of NH_2 group, appeared at 6.71 ppm are disappeared when synthesized compounds are cyclized, one proton of NH-CH appeared at 11.29 ppm disappeared, whereas NH-CO proton band shifted and appeared as a singlet at 8.81 ppm, shown by Ali et al. in 2018. Aromatic protons increases in synthesized compounds ^{13}C-NMR spectra of Synthesized compounds support the formation of new compounds after cyclization.

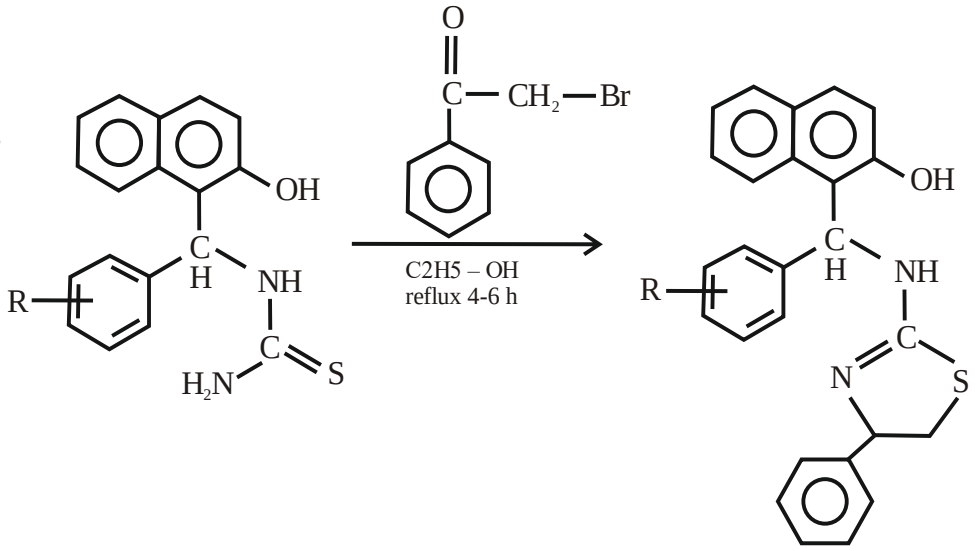
The chemical shifts for carbon of C = S group in compounds are disappeared and the carbon of C = $N_{thiazole}$ group are formed at different values in ppm, whereas carbon of C – $N_{thiazole}$ are appeared at 153-163 ppm and 118-120 ppm respectively, this shows number of carbon atoms increases, when synthesized products are formed, because of the presence of the carbon CH_2, C = $O_{carboxylic\ acid}$ C – $S_{thiazoledin\text{-}4\text{-}one}$, C = $O_{thiazoledin\text{-}4\text{-}one}$ and C – $N_{thiazoledin\text{-}4\text{-}one}$ at 177.95, 48.10, 56.55, 169.57 and 145.78 ppm respectively. Carbon of C = S at 183.67 ppm are disappeared. All different synthesized compounds shows the anti-microbial activity, which inhibits the growth of micro-organisms or kills the micro-organisms.

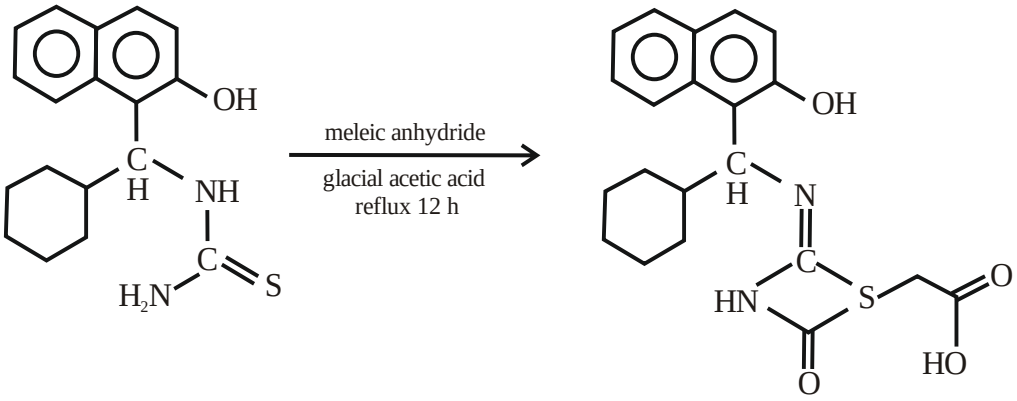
When compounds are changed into heterocyclic compounds their anti-bacterial and anti-fungal activities are increases. Synthesized compounds was highly active while prepared as 800-1200 µg/ml.

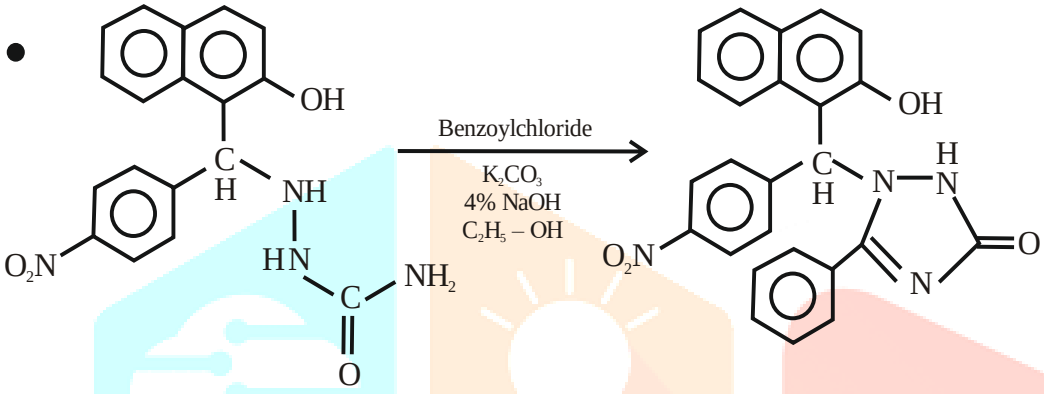
Synthesis of Various Compounds :

- 

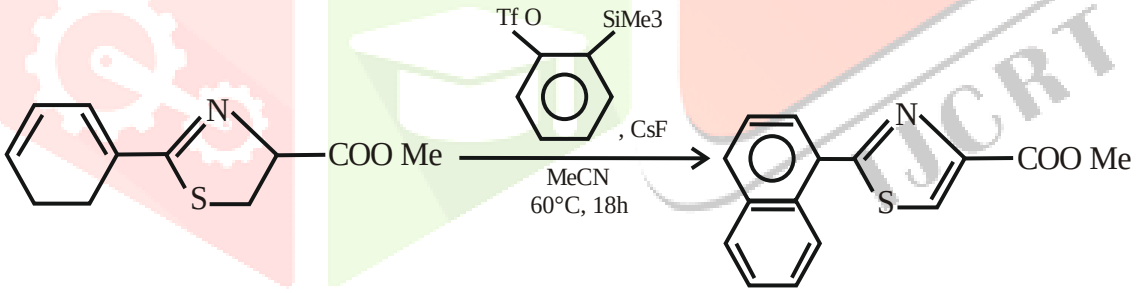
- 

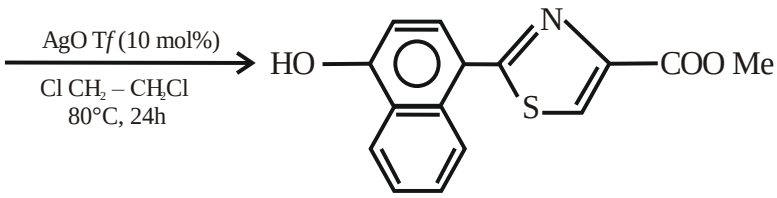
- 

- 

- 

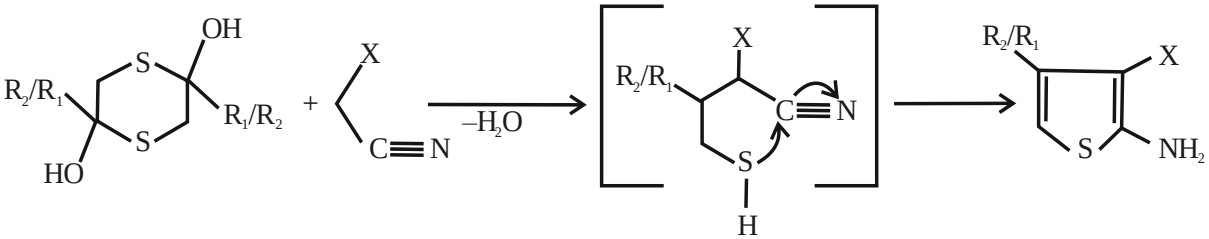
All synthesized compounds shows that anti-bacterial and anti-fungal properties at proper concentrations. Both Gram positive and Gram negative species are affected, whereas Candida species affected in fungus group.





Synthesis of thiazole-conjugated naphthol

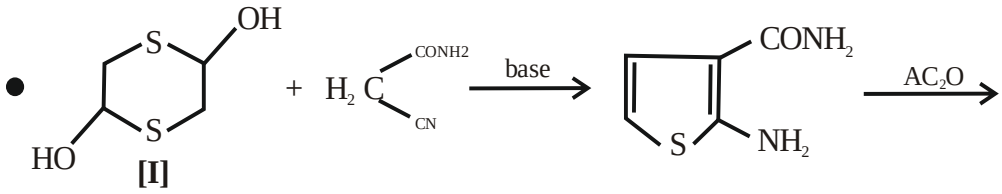
Heterocyclic Organic Synthesis :

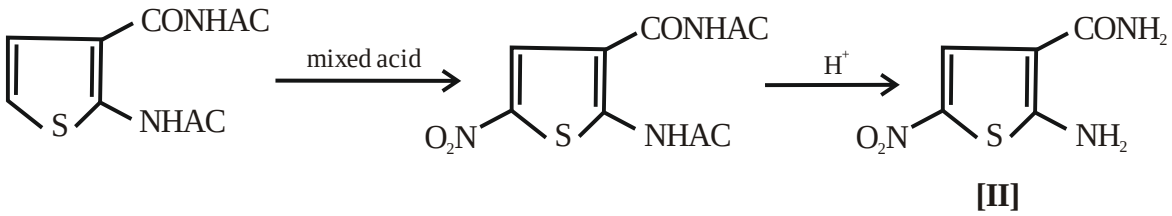


Where, R_1/R_2 = H, Me

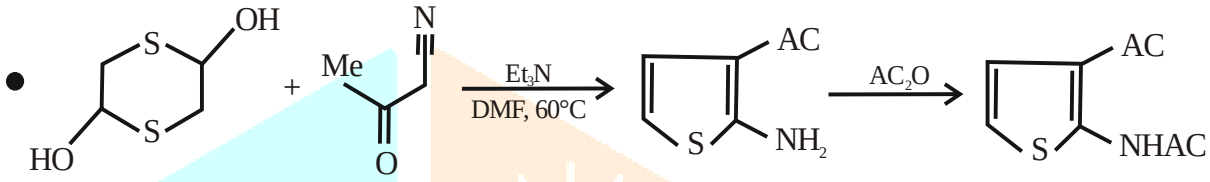
X = CN, Me, Co_2Me, $CONH_2$

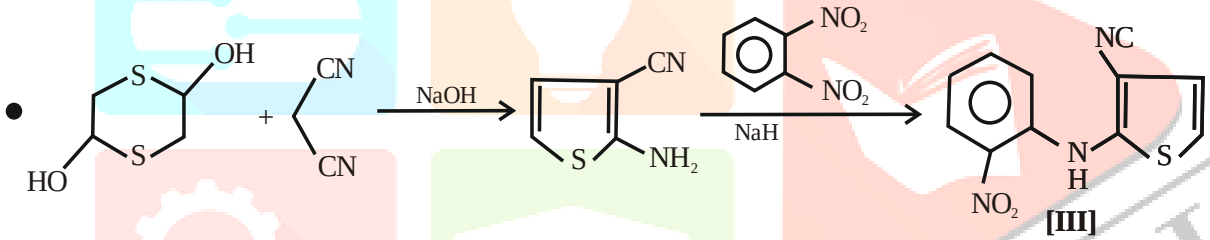
Gewald Reaction :



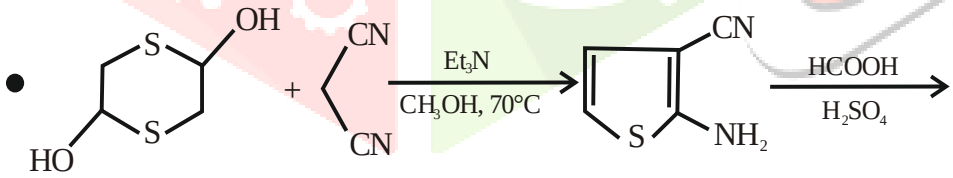


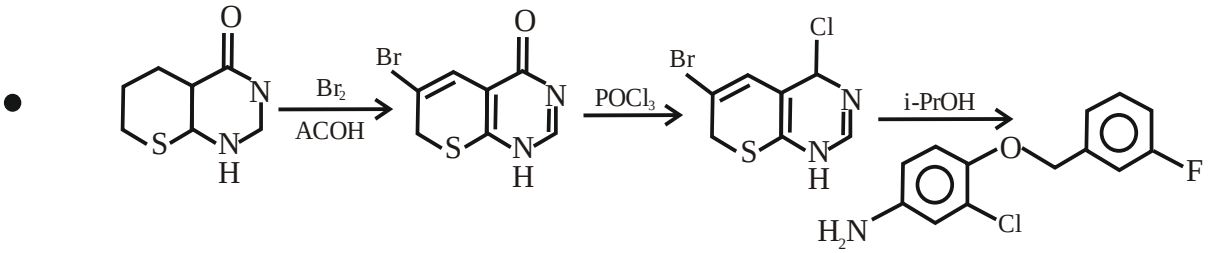
[II]

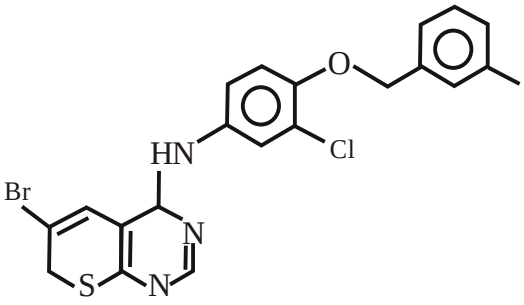




[III]







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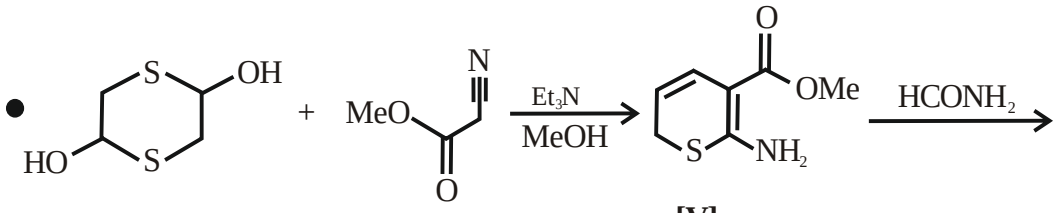
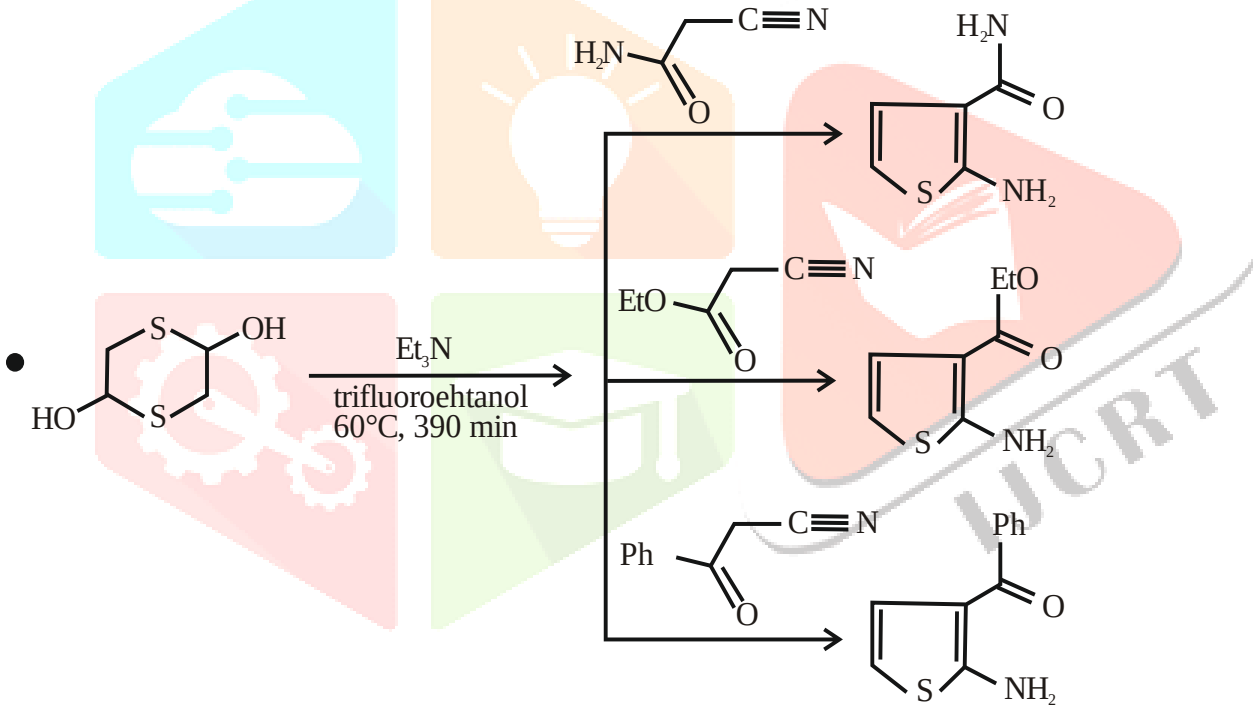
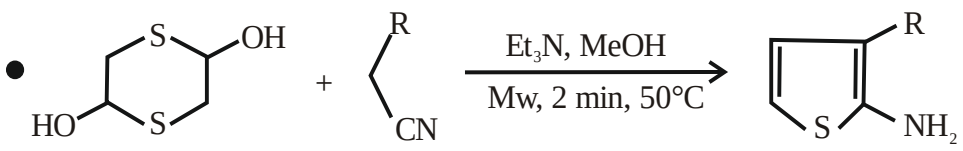


[V]





[VII]

Where, R = CO, COOMe, $CONH_2$, CONHPh, CO_tBu

Conclusion :

In this study, the simple and useful methods are used to synthesized new heterocyclic compounds and their derivatives in good quantities nitro group needs less reaction time than hydrogen. TLC method was used to monitoring, while ultrasonic technique is used to save time. Ethanol is common suitable solvent that used for recrystallization the synthesized products. heterocyclic compounds and their derived synthesized compounds acts as anti-microbial activities. These compounds shows potent anti-bacterial and anti-fungal activities. Growth of micro-organism decreases with the concentration of synthesized compounds increases.

In this review, we conclude that heterocyclic compounds and their derivatives, are very important in the biological point of view as well as medicinal point of view, hence, their study, synthesized and uses are necessary to the wellness of human beings.

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