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Synthesis of triazoles via click chemistry approach through the preparation of a recyclable hydrotalcite supported copper nano catalyst.

VAIBHAV TRIPATHI[a*], AMULYA SINHA[b] , ARZOO SIDDIQUI[b], AMIT YADAV[b],GIRISH SINGH[b], DHARMENDRA SINGH[c] and VIVEK BHADAURIA[a].

[a*,a,b] Green Laboratory, Department of Chemistry, Ewing Christian College (University of Allahabad), Prayagraj-211003, India.

[c]Bioorganic Lab,Department of Chemistry,Institute of Science,Banaras Hindu University,Varanasi-221005, India.

Abstract: Heterocyclic chemistry serves as an illustration for the lack of clear boundaries because it permeates many of the other chemical fields. Biological processes are intimately associated with heterocycles. Heterocycles are very important to the pharmaceutical and agricultural industries due to their natural occurrence. Heterocycles and the chemistry-biology link are present in about 90% of newly developed drugs. Triazole, also known as pyrrodiazole, has a nitrogenous heterocyclic component and has the chemical formula $C_2H_2N_3$. Many studies have been conducted on five-member heterocyclic triazole rings. The pharmacological activities of triazole compounds include antibacterial,anticonvulsant, antitubercular, antituberculosis, antimicrobial, and anti-inflammatory qualities.

In order to determine the molecular structure, substituted triazoles containing heterocyclic scaffolds have been synthesised and characterised using NMR and MS spectroscopy. Using hadrotalcite as a solid support and water-ethanol as an effective solvent, a one-pot multicomponent mechanistic pathway in the presence of Cu nanocatalyst was used to carry out the synthesis of this method.

Keywords:Triazole derivatives,Multicomponent Reactions,Azide Alkyne Reactions(AAC),CuNPs.

Introduction: In order to create important target heterocyclic compounds in a single step, organic synthesis has recently seen the development of efficient technologies and environmentally friendly solvents for organic transformations. This is now a common objective for both pharmaceutical corporations and researchers. Multicomponent reactions (MCRs) are becoming a common tool in synthetic organic, combinative chemical research, and drug development processes.[1-8]

Excellent yields, atom economy, high peculiarity, fast reaction times, and resilience to potentially hazardous reagents and purification processes are just a few of MCRs' many notable benefits. With the advent of combinatorial processes in MCRs, the development of new compounds and procedures experienced a standard shift. Today, the primary concerns are sustainability in the environment, affordability, atom efficiency, and variability.One crucial component of sustainable chemical processes is the use of environmentally friendly solvents.[8-12]

The use of heterogeneous catalysts in organic synthesis has garnered a lot of attention. According to Roy and colleagues, 1,4-disubstitutedrate 1,2,3-triazole derivatives of azides produced in situ from anilines or amines and terminal acetylene in aqueous media can be synthesised in a single pot using polystyrene-anchored Cu(II) as a catalyst.

Chemistry plays a major role in research and the development of novel bioactive substances. The identification and development of the important physiologically active pharmacological molecule is the focus of medicinal chemistry, a branch of the pharmaceutical and medical sciences. These heterocyclic compounds of nitrogen and oxygen have been found to have the most biological activity. Because of their successful application in medicinal chemistry, azolic derivatives such as thiazole, triazole, oxadiazole, and thiadiazole are pharmacologically active substances. Triazole, also known as pyrrodiazole, is a five-member heterocyclic ring with the chemical formula $C_2H_2N_3$. It has wo isomeric forms: 1,2,3-triazole and 1,2,4-triazole. It has two carbon atoms and three nitrogen atoms.

Two double-bonded carbon atoms and two normal nitrogen atoms make up the 1,2,3-triazole, an unsaturated, aromatic, five-membered, p-excessive nitrogen heterocycle with a 6p electron ring structure. One pyrrole type and two pyridine types make up the total of three nitrogen atoms. Using a heterogeneous Cu nanocatalyst, the azide-alkyne cycloaddition (AAC) reaction—which is based on the click chemistry approach—is employed to synthesise triazole. Since it is the foundation of the best modern fungicides for agriculture and medications for fungal illnesses in humans, 1,2,4-triazole is more significant.

A modular synthetic method for creating new chemical entities is called "click chemistry." This potent tactic primarily depends on the use of spring-loaded reactants to create carbon–heteroatom bonds. It has an increasing number of uses in almost every branch of contemporary chemistry, from materials science to medication development. Because of its dependability, specificity, and biocompatibility, the copper(ll)-catalyzed 1,2,3-triazole forming reaction between azides and terminal alkynes has emerged as the gold standard in click chemistry.

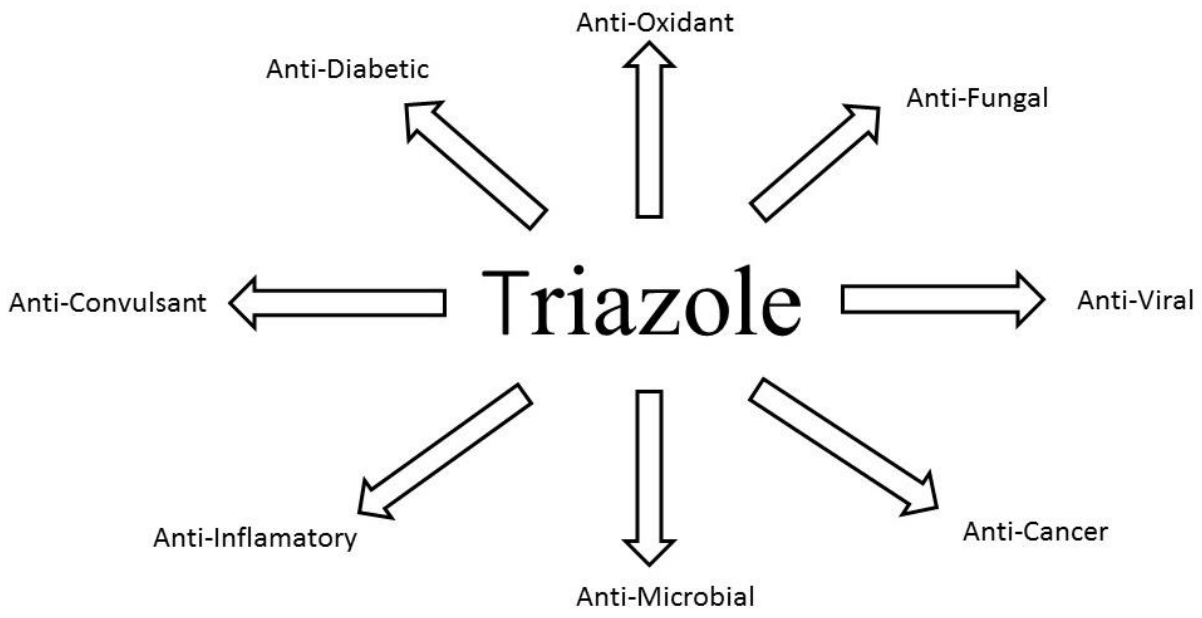


Fig.1:The triazole scaffold has significant biological activity.

Experimental Section:

Matrial & Methods: Commercial sources provided analytical-grade chemicals and reagents for the procedure, which were used without the need for further purification. All of the first reagents, including hydroxylamine hydrochloride and ethyl acetoacetate, were purchased from TCI and Sigma-Aldrich. The melting points were determined using the uncorrected open glass capillary technique.

The BRUKER AVANCE II 400 FT spectrophotometer (1H NMR at 400 MHz and 13C NMR at 100 MHz) was used to analyze the structure identification for the synthesized target moeity.Using TMS as the internal standard and $CDCl_3$ as a solvent, the chemical shift results are expressed as δ(ppm). The tests were observed using twenty 0.25 mm thick silica gel G/UV-254 pre-coated TLC sheets. At 70 eV, JEOL SX-102 (FAB) was used to analyze the mass spectra.

Preparation Method of the Catalyst: Hadrotalcite with formula $Ca_6Al_2(CO_3)(OH)_{16}.4H_2O$.

Hydrotalcite is synthesised by co-precipitation. In this process, two solutions were prepared one of them is a mixture of 2M NaOH, 0.2M Na_2CO_3 & Second solution Contained Ca^{+2} as $CaCl_2$ with 0.75M Concentration together with 0.25M Al^{+3} ($AlCl_3.6H_2O$).

Then,adding second solution in solution first one dropwise under vigorous stirring. Now,precipitate washed & dried in an oven (85 degree temp.) for 8hour.

Preparation of Cu@HT nanocatalyst: $CuSO_4.5H_2O$ (0.5gm) was used to disperse hadrotalcite in 500ml of water at room temperature for 30 minutes. After adding 50% hydrazine hydrate (10 ml) dropwise for 30 minutes, the reaction mixture was agitated for two hours at room temperature.
The resulting solid is next centrifuged using acetone and distilled water, and it is dried for eight hours at 100 degrees.

Reaction Procedure: Reaction Process is In-Situ Reaction in which 1 milimole of Alkyne,1 milimole of Alkyl Halide,Solvent(Water-Ethanol)4ml,Sodium L-Ascorbate(0.02gm) & varying amount of catalyst(10 to 20 mg) are added together in a R.B.Flask then the reaction process is occurred with the help of magnetic stirrer at 50ºc temperature.

After Several hours some soil or yellowish colour products are obtained.

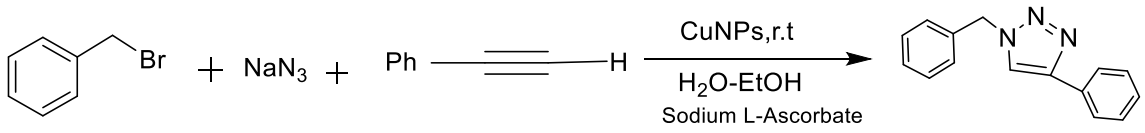
After that We check TLC(Thin layer Chromatography) of these compounds in which very small amount of product yield taken in a TLC beaker and added some amount of n-hexane & some amount of chloroform one by one(At first 2:2 then 2:3).

Compound is dissolved then with the help of TLC plates & Capillary tube the Thin-Layer Chromatography process is done.

After that a clear single spot obtained.

Result & Discussion:

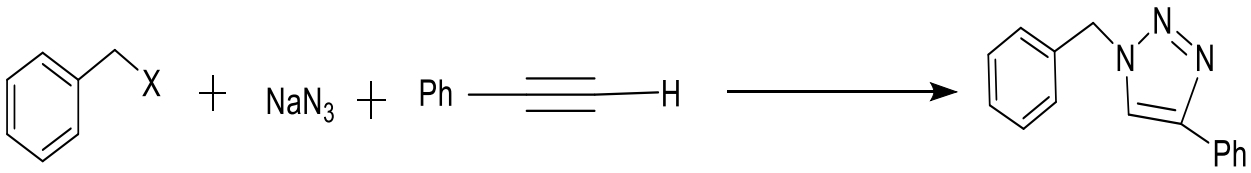
Table 1: Solvent Study

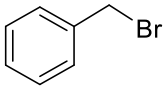
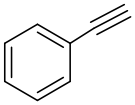
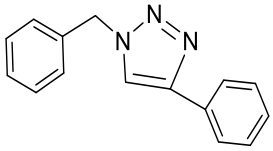
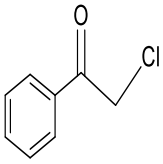
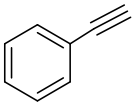
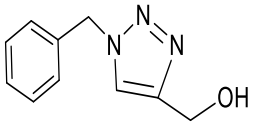
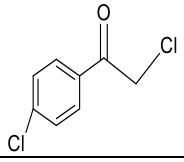
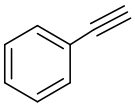
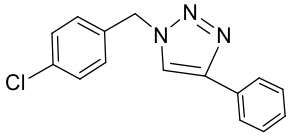
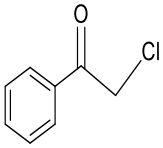
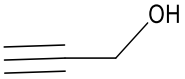
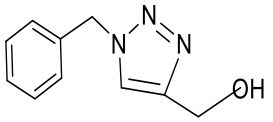
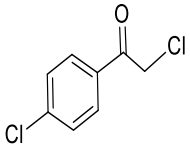
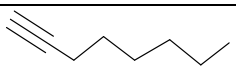
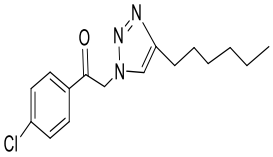
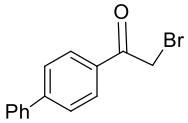
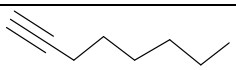
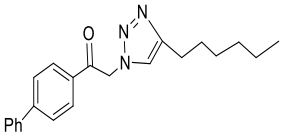
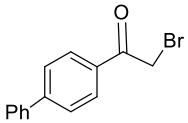
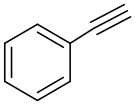
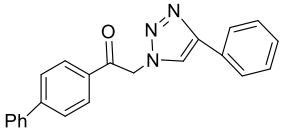


S.N.	Catalyst	Amount(in mg)	Condition	Temperature (°c)	Time	Yield
1	CuO	15	H_2O/EtOH(1:1)	50	8h	92%
2	CuO	20	H_2O/EtOH(1:1)	50	8h	83%
3	CuO	10	H_2O/EtOH(1:1)	50	10h	74%
4	CuO	15	H_2O/EtOH(3:1)	50	14h	68%
5	CuO	5	H_2O/EtOH(1:1)	50	18h	56%
6	CuO	15	H_2O/EtOH(1:3)	50	14h	52%
7	CuO	15	Ethylene Glycol	50	20h	58%
8	CuO	10	Ethylene Glycol	50	22h	55%
9	CuO	15	H_2O/EtOH(1:1)	r.t	17h	45%
10	CuO	10	H_2O/EtOH(1:1)	r.t	18h	42%
11	----	----	H_2O/EtOH(1:1)	50	24h	38%
12	----	----	Neat	50	24h	32%

Reaction Condition: Benzyl Bromide(1mmol),Sodium Azide(1mmol), Phenyl Acetylene(1mmol),Solvent(4ml),Sodium L-Ascorbate(0.02gm).

Table 2: Click Reaction of Primary Halides with terminal alkynes(Substrate Scope):



Entry	Aryl halide	Alkyene	Time	Triazole	Yield(%)
1.			8h		92
2.			8h		84
3.			10h		81
4.			12h		82
5.			16h		67
6.			13h		75
7.			12h		73

Reaction Condition:Aryl Halide(1mmol), Sodium Azide(1mmol),Alkyne(1mmol),Solvent(4ml),Sodium L-Ascorbate(0.02gm).

Characterisation of Catalyst:- By X.R.D

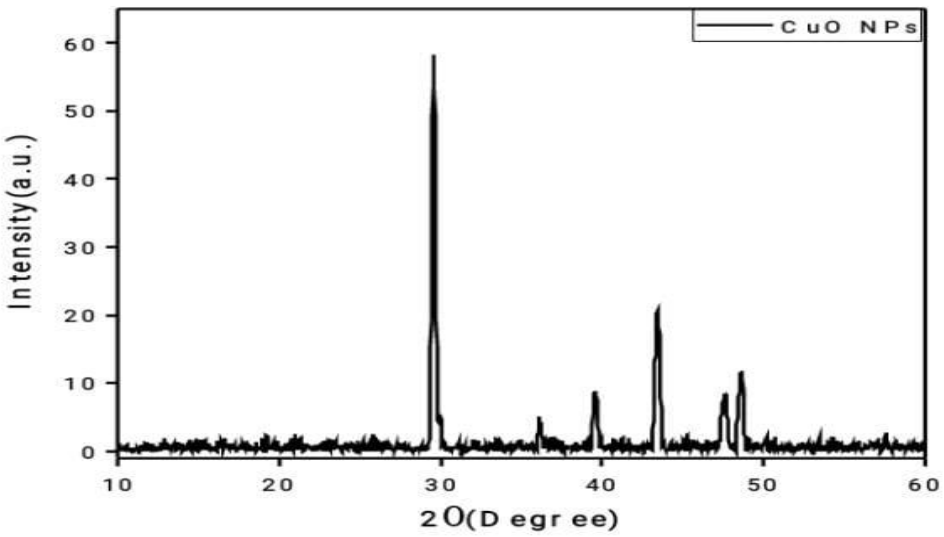


Fig.2

An X-ray diffraction (XRD) pattern of CuO nanoparticles (CuO NPs) is shown in the graph. It displays the correlation between diffraction intensity and diffraction angle (2θ), which is used to determine the material's phase, crystallinity, and crystal structure.
The diffraction angle (2θ) in degrees is represented by the horizontal X-axis. The diffraction intensity is shown on the vertical Y-axis in arbitrary units (a.u.).
The produced CuO nanoparticles are highly crystalline, as seen by the XRD pattern's multiple distinct diffraction peaks. CuO nanoparticles predominant crystallographic orientation is indicated by the highest peak at 2θ = 35–39°. Good crystallinity, wider crystalline domains, and fewer structural flaws are indicated by the sharp and narrow peaks.

Spectral Data:

1):- 1-benzyl-4-phenyl-1H-1,2,3-triazole:

m.p.- 114°c

^{1}HNMR:(400MHz,$CDCl_3$),∂_H(ppm);

5.48(s,2H,CH_2),7.34(t,H,CH_{Ar}),7.49(d,1H,$^{3}J_{HH}$=7.4Hz,2CH_{Ar}),7.53(t,2H,$^{3}J_{HH}$=8.2Hz,2CH_{Ar}), 7.84(d,2H,$^{3}J_{HH}$=7.4Hz,2CH_{Ar}),**7.65**(s,1H,CH),7.68(t,2H,$^{3}J_{HH}$=8.2Hz,2CH_{Ar})

^{13}C NMR:-100MHz,$CDCl_3$,∂_C(ppm);

54.8,118.41,123.6,126.8,127.6,128.2,**129.6**,131.7,136.8,138.6,143.4.

2)-1-benzyl-4-phenyl-1H-1,2,3-triazole:

m.p.- 118°c

^{1}H NMR:(400MHz,$CDCl_3$),∂H(ppm);-

5.64(s,2H,CH_2),7.49(t,2H,CH_{Ar}),7.56(t,2H,$^{3}J_{HH}$=7.6Hz,2CH_{Ar}),7.68(d,2H,$^{3}J_{HH}$=8.4Hz,2CH_{Ar}), 7.89(d,2H, $^{3}J_{HH}$=7.6Hz, 2CH_{Ar}),7.73(d,2H, $^{3}J_{HH}$=8.4Hz).

^{13}C NMR:- 100MHz,$CDCl_3$,∂_C(ppm);
55.6,118.2,124.6,127.2,128.4,129.3,130.1,132.3,137.2,139.4,144.1,190.4(C=O).

3)- (4-chlorophenyl)(4-phenyl-1H-1,2,3-triazol-1-yl)methanone:

m.p.- 117°c

1H NMR:(400MHz,$CDCl_3$),∂H(ppm);-

5.92(s,2H,CH2),7.54(t,1H,m,CHAr),7.48(t,2H,3JHH=7.6Hz,2CHAr);7.52(d,2H,3JHH=8.2Hz,2CHAr), 7.85(d,2H, 3JHH=7.5Hz, 2CHAr),7.93(s,1H,CH),7.96(d,2H,3JHH=8.2Hz,2CHAr)

13C NMR:- 100MHz,$CDCl_3$,∂C(ppm);

55.2,121.21,125.6,128.1,128.2,129.4,130.2,131.6,138.4,141.6,148.4,189.4(C=O),

4)- (4-(hydroxymethyl)-1H-1,2,3-triazol-1-yl)(phenyl)methanone:

m.p.- 121°c

^{1}H NMR:(400MHz,$CDCl_3$),∂H(ppm);

2.35(broad,s,1H,OH),4.86(s,2H,CH_2),5.82(s,2H,CH_2),7.40(d,2H,$^{3}J_{HH}$=8.6Hz,2CH_{Ar}),7.72(s,1H, CH), 7.95(d,2H, $^{3}J_{HH}$=8.6Hz,2CH_{Ar}).

13C NMR:- 100MHz,$CDCl_3$,∂C(ppm);

55.2,56.4,123.2,129.1,129.4,132.4,140.6,146.9,189.4

5)- 1-(4-chlorophenyl)-2-(4-hexyl-1H-1,2,3-triazol-1-yl)ethan-1-one:

m.p.- 108°c

^{1}H NMR:(400MHz,$CDCl_3$),∂H(ppm);-

0.90(t,3H,$^{3}J_{HH}$=7.2Hz,CH_3),1.32(m,2H,2CH_2),1.32(m,2H,CH_2),1.68(m,2H, $^{3}J_{HH}$=7.4Hz,CH_2), 2.76(t,2H, $^{3}J_{HH}$=7.6Hz,CH_2),5.86(s,2H,CH_2),7.42(s,1H,CH,),7.56(d,2H,$^{3}J_{HH}$=8.4Hz,2CH_{Ar}), 7.90(d,2H,$^{3}J_{HH}$=8.4Hz,2CH_{Ar})...

^{13}C NMR:- 100MHz,$CDCl_3$,∂C(ppm);14.1,22.4,25.6,27.8,29.2,30.6,55.0,122.1,129.2,129.2,130.6,140.6,148.6,189.2(C=O).

6)- 1-([1,1'-biphenyl]-4-yl)-2-(4-hexyl-1H-1,2,3-triazol-1-yl)ethan-1-one:

m.p.- 121°c

^{1}H NMR:(400MHz,$CDCl_3$),∂H(ppm);-

0.87(t,3H,$^{3}J_{HH}$=7.4Hz,CH_3),1.35(m,4H,2CH_2),1.37(m,2H,CH_2),1.71(2H, $^{3}J_{HH}$=7.4Hz,CH_2), 2.77(t,2H, $^{3}J_{HH}$=7.7Hz,CH_2),5.43(s,2H,CH_2),7.56(s,1H,CH,), 7.67(d,2H, $^{3}J_{HH}$=8.2Hz,2CH_{Ar}),7.96(d,2H,$^{3}J_{HH}$=8.2Hz,2CH_{Ar})

^{13}C NMR:- 100MHz,$CDCl_3$,∂C(ppm);

14.6,22.1,25.9,27.8,29.6,30.3,55.2,122.6,127.2,126.3,130.9,141.8,148.3,191.6(C=O).

7)- 1-([1,1'-biphenyl]-4-yl)-2-(4-phenyl-1H-1,2,3-triazol-1-yl)ethan-1-one:

m.p.- 112ºc

^{1}H NMR:(400MHz,$CDCl_3$),∂H(ppm);-

5.93(s,2H,CH_2),7.32(t,1H, $^{3}J_{HH}$=7.3Hz,CH_{Ar}),7.48(m,3H,3CH_{Ar}),7.43(t,2H, $^{3}J_{HH}$=7.3Hz,2CH_{Ar}), 7.37(t,2H,$^{3}J_{HH}$=7.3Hz,2CH_{Ar}),7.78(t,2H,$^{3}J_{HH}$=8.1Hz,2CH_{Ar}),7.97(d,2H,$^{3}J_{HH}$=7.3Hz,2CH_{Ar}), 8.02(s,1H,CH), 8.18(d,2H, $^{3}J_{HH}$=8.3Hz,2CH_{Ar}).

^{13}C NMR:- 100MHz,$CDCl_3$,∂C(ppm);

55.4,123.4,125.6,127.6,127.7,128.1,128.8,128.3,128.9,129.3,130.6,132.5,139.2,147.4,148.2,189.6(C=O).

Mass Spectroscopy:-

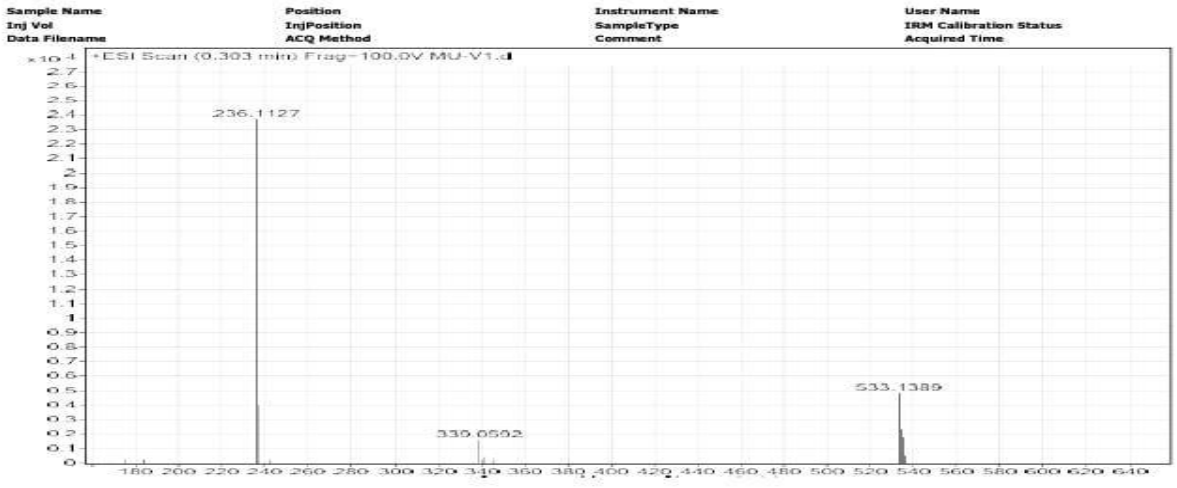


Fig.3

It is an Electrospray Ionisation Mass Spectrometry (ESI-MS) spectrum that uses the mass-to-charge ratio (m/z) to identify chemicals.

• X-axis (m/z): Shows the discovered ions' mass-to-charge ratio (m/z).

• Y-axis (Intensity): Shows each ion's relative abundance (signal intensity).

Greater concentrations of ions are indicated by higher peaks.
Value of m/z (236.1127) The most prevalent ion, base peak (highest intensity), is probably the most stable fragment or the molecular ion $[M+H]^+$. The molecular weight of the molecule is roughly 235.11 Da if it is the protonated molecular ion. The base peak, which has the highest intensity (100% relative abundance), is located at m/z = 236.1127. This suggests that this ion is the most prevalent and stable species found in the mass spectrum.
According to the spectrum, there is just one significant molecule in the sample under analysis, which produces a dominating ion at m/z 236.1127.

Recylability of the Catalyst: All reactions were carried out using Azide(1mmole), Alkynes(1mmol),Catalyst(15mg),Sodium L-Ascorbate(0.02gm) in Water-Ethanol(4ml) at 50ºc to give isolated yields.

The catalytic system's recyclability and reusability are used to gauge a heterogeneous catalyst's effectiveness.The same catalytic system was used to conduct the reaction between bynzyl azide and phenyl acetylene up to the fifth time without significantly reducing catalytic activity. Azide Alkyne Cycloaddition (AAC) reactions were conducted using a 15 mg catalyst in water-ethanol at 50ºC. Following each run, the catalyst was recovered by centrifugation and the product was extracted using ethyl acetate from water ethanol.

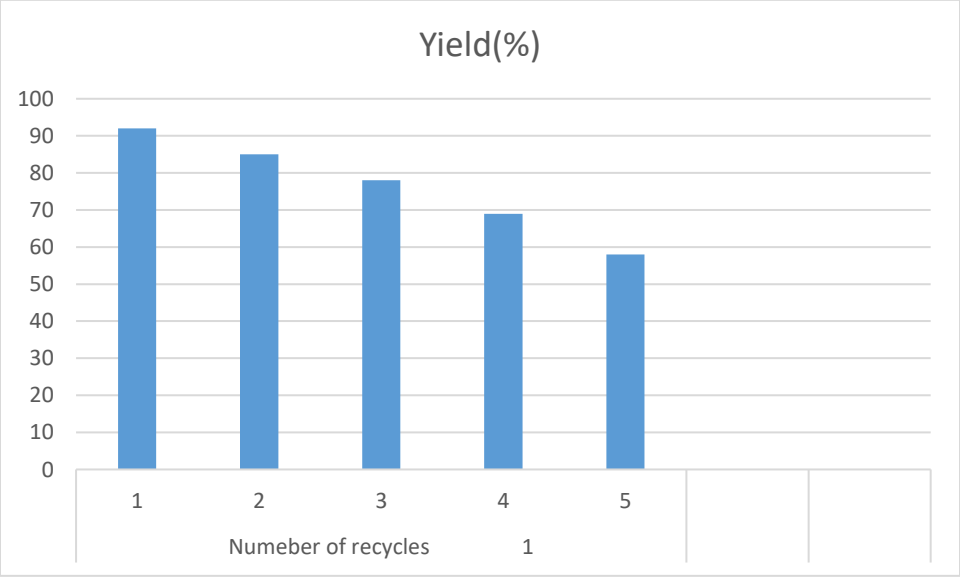


Fig.4: (Recyclability of the Catalyst)

Conclusion:

We have developed a highly efficient, recyclable, and reusable heterogeneous catalyst for the important synthesis of triazole derivatives via the cycloaddition of azides and alkynes. This method accommodates a wide range of azides and alkynes, producing pharmaceutically significant 1,2,3-triazoles. The use of benign water-ethanol & ethylene glycol as the solvent and the reaction proceeding at room temperature make this an environmentally friendly system.

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Conflict of Interest:- The authors declare that there is no conflict of interests regarding the publication of this article.

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