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Fabrication of Zr-based Metal Organic Framework and Its Application in to Photocatalysis and Electrocatalysis by Dye Removal Studies

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Abstract - In this work, the photocatalytic degradation of Acid Yellow, 99 (AY 99) dye under solar light irradiation using the Zr-MOF@GNS and synthesizedZrO_2isinvestigated.TheZrO_2issynthesizedusingZr-metalprecursor. The prepared ZrO_2 is used to synthesize Zr-MOF@GNS and it is further examined using FT-IR, PXRD and FESE Manalyses. The photocatalyticdegradationofAY99 is studied by varying the initial concentrationofAY99, the length of the irradiation period, the catalyst dose and the pH of the dye solution. From the result sit is found that the Zr-MOF@GNS is an efficient and promising catalyst for the enhancement ofphotocatalyticdegradationofAY99.Electrochemicalstudiesshowsthat$ZrO_2$and Zr-MOF@GNS can act as electrocatalyst for acid base titrations.

Keywords : ZrO_2Zr-MOF@GNS, AY99, Photocatalysis and Electrocatalysis,.

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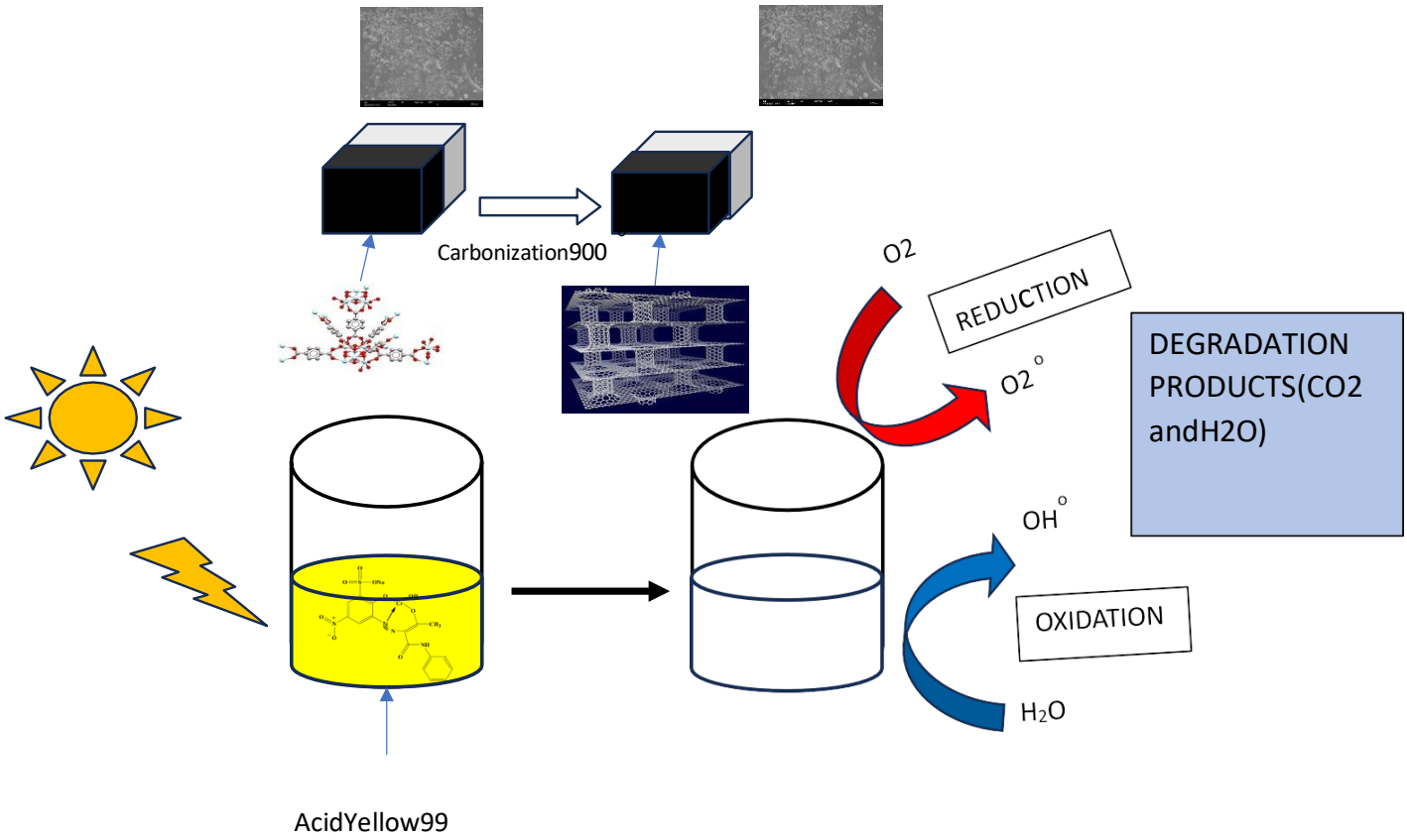
Graphicalabstract



INTRODUCTION:

ThetoxicityandenvironmentalpersistenceofMBposeseriousrisks:“Oneofthe popular acidic dyes that is environmentally persistent, toxic, carcinogenic and mutagenic isAcidYellow 99[01]. Health effects range from respiratory distress, tissue necrosis, and jaundice to oxidative damage, due to MB’s monoamine oxidase inhibition and its water solubility. MB also enters food chains, impairing aquatic life and posing bioaccumulation risks [02]

Acid Yellow 99 (AY-99) is an azo dye commonly used in the textile and leather industries for its vibrant yellow color. The chemical structure of AY-99, which includes an azo bond (-N=N-) and aromatic rings, makes it highly resistant to biodegradation and other conventional removal methods. The persistence of such dyes in aquatic environments poses significant ecological and health risks.AY-99, like many azo dyes, is toxic to aquatic organisms and can disrupt ecosystems by decreasing oxygen levels in water bodies [3]. The coagulation process effectively decolorizes insoluble dyes, but it fails to work well with soluble dyes. Traditional methodsforremovingdyesfromwastewaterincludeadsorption,chemicaloxidation, biological treatment and membrane filtration [4]. However, these techniques often come with limitations such as incomplete dye removal, generation of secondary pollutants, or high operational costs. are effective but tend to be energy-intensive and may generate toxic by-products [5]. Biological treatments, while environmentally friendly,may notbeeffectiveforrecalcitrant dyeslikeAY-99 due to their complex molecular structure and resistance to microbial degradation. The long-term presence or accumulation of these dyes in waste water discharged from these industries is detrimental to the aquatic environment. Given these challenges, theneedformoreefficient,sustainable,andeco-friendlymethodstodegradeorganic pollutants, including dyes, is crucial [6]. Synthetic dyes are widely used in various industries such as textiles, leather, paper, plastics, and cosmetics, leading to the discharge of large volumes of dye-containing effluents into water bodies. These dyes, particularly **methylene blue**,

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crystal violet, and **rhodamine B,Acid yellow 99** are not only toxic and carcinogenic but also resistant to biological degradation due to their complex aromatic molecular structures. Hence, effective and eco- friendly strategies for dye removal are essential for environmental protection.

Among several advanced oxidation processes, **photocatalytic degradation** has emergedasahighlypromisingmethodfordyeremovalduetoits**cost-effectiveness, reusability,andabilitytocompletelymineralizedyesunderlightirradiation**.

This process typically utilizes **semiconductor photocatalysts** (e.g., TiO_2, ZrO, g- C_3N_4) that, under UV or visible light, generate electron-hole pairs capable of producing reactive oxygen species (ROS) such as hydroxyl radicals (•OH) and superoxide radicals ($•O_2^-$), which attack and break down dye molecules into non- toxic end products like CO_2 and H_2O.

KeyFeaturesandEfficiencyofPhotocatalyticDegradation

- **Eco-friendlyandnon-selective**:Candegradeawiderangeoforganic pollutants without the need for additional chemicals.
- **Lowenergyrequirement**:Especiallyundervisiblelightorsunlight,making it suitable for sustainable applications.
- **No secondary pollution**: Unlike adsorption or chemical treatments, photocatalysis does not produce harmful residues.
- **Regenerability**: Photocatalysts can be reused multiple times with minimal loss in activity [7].

Recent studies have shown enhanced performance through **doping**, **composite formation**, and **nanostructuring** of photocatalysts. For instance, **Ag-doped ZnO nanocomposites** demonstrated superior visible-light-driven degradation efficiency for methylene blue dye, attributed to better charge separation and light absorption [8].

Photocatalyticdegradationoffersaviablesolutionbyutilizinglightenergytodrive chemical reactions that break down harmful pollutants into non-toxic by-products, suchaswaterandcarbondioxide[9].However,theapplicationofphotocatalysisfor environmental remediation is still challenging task.

Metal-organicframeworks (MOFs) are aclassofmaterials consistingofmetal ions or clusters coordinated to organic ligands, forming a highly porous, crystalline structure.MOFshavegainedsignificantattentionasphotocatalystsduetotheirhigh surface area, tunable structure, and ability to adsorb a wide variety of organic pollutants [10]. metal−organic framework (MOF) possesses high surface area, chemical tunability, and abundant active adsorption sites, resulting in MOF adsorbents exhibit high adsorption capacity for metal ions. These properties make MOFs ideal candidates for applications in environmental remediation, including photocatalyticdegradationofdyes[11,12].AmongvariousMOFs,zirconium-based MOFs(Zr-MOFs)haveshownexceptionalpromiseduetotheirhighstability,large surfaceareaandabilitytoenhancephotocatalyticperformance.commercial

monoclinicZrO_2,akeycomponentinZr-MOFs,isknownforitsexcellentchemical

stability, thermal resistance, and low toxicity. These properties make ZrO_2-based MOFs highly suitable for photocatalytic applications, especially in harsh environments where other photocatalysts might degrade or lose activity. Zr-MOFs are also highly stable in aqueous environments, a critical factor for wastewater treatment processes [13-15].

,**Metal-OrganicFrameworks(MOFs)**haveemergedasoneofthemostpromising classes of photocatalysts for the degradation of organic dyes from wastewater. Due totheir**highsurfacearea,tunableporosity,structuraldiversity,andphotoactive metal centers**, MOFs offer superior photocatalytic performance compared to conventional semiconductors like ZrO. MOFs such as **MIL-125(Ti)**, **UiO-66(Zr)**, and **NH_2-MIL-88B(Fe)** exhibit excellent light absorption and charge separation capabilities, facilitating the generation of reactive oxygen species (ROS) under UV and visible light [16]. Furthermore, the integration of light-harvesting linkers (e.g., amino-terephthalate) into the MOF structure enhances the absorption in the visible region,makingthemidealforsolar-drivendyedegradation.Additionally,MOFscan be **post-synthetically modified** or composited with other materials like graphene oxideornoblemetalstofurtherenhancephotocatalyticefficiencyandstability[17]. Notably, studies have shown that MOF-based photocatalysts exhibit high degradation efficiency toward common dyes such as **methylene blue**, **rhodamine B**, and **crystal violet**, achieving nearly complete mineralization without generating toxic byproducts. In addition, the crucial electronic and/or surface structures of hollow and/or porous nanostructures can be tuned with the use of MOFs, thereby promotingelectrocatalyticactivity[18].Thepresentstudyfocusesonphotocatalytic applications of ZrO2 and Zr-MOF@GNS usingAY99 dye as a model compound. The electro chemical studies done using strong acid and

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strong base titrations.

2. EXPERIMENTAL

Materials

TheAY99dyeispurchasedcommerciallyfromlocalvendors($C_{25}H_{19}N_4NaO_8S_2$, CAS:10343-58-5). De-ionized water used in the process. Zirconium oxychloride is procured from Nice chemicals (P) Ltd. and all other reagents are used as such without further purification.

Synthesisof ZrO2

ZrO_2issynthesizedbymixing0.1MofNaOHwitha 2mMsolutionofmelamine and 2.5 mM of zirconium oxychloride in a magnetic stirrer. The loosely bonded metal residue is baked in a muffle furnace at 900^0C for 12 hours to produce white powder after being repeatedly cleaned by centrifugation with alcohol.

SynthesisofZr-MOF@GNS

Inordertoaccomplishcarbonization,thesampleisplacedinamicrowaveovenand heated to around 500^0C for three hours while being exposed to oxygen.To oxidise theZrO_2,thesampleisallowedtocooltoambienttemperaturebeforebeingannealed for an hour at 300^0C in an air oven.

2.3Conductometricdetermination

TheconductometrictitrationofStrongacidandstrongbaseiscarriedout usingZrO2andZr-MOF@GNSasaelectrocatalyst0.1NofHClistitratedwith0.5N NaOH solution by adding 0.005g/l of Zr-MOF@GNS. The values observed are plottedagainstwhenthevolumeofNaOHisaddedfromthecurveobtainedtheend point is determined graphically.

3. RESULTANDDISCUSSION

Characterization

The synthesized Zr-MOF@GNS and ZrO_2 are examined using FT-IR, FESEM and PXRD analyses. The surface morphology of ZrO_2 and Zr-MOF@GNS is characterizedusingascanningelectronmicroscope(SEM,HitachiS-4800).Fourier Transform Infrared Spectroscopy (FT-IR) measurements are carried out with a SHIMADZU IRTRACER 100, utilizing the KBr pellet method over a range of 4500–500 cm^{-1}.The crystalline phase structure is confirmed viaPowder X-ray Diffraction(PXRD) using a BRUKER USA D8 Advance DaVinci diffractometer with Cu-Kα radiation, scanning from 0° to 100°. [19-20]

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FT-IRAnalysis

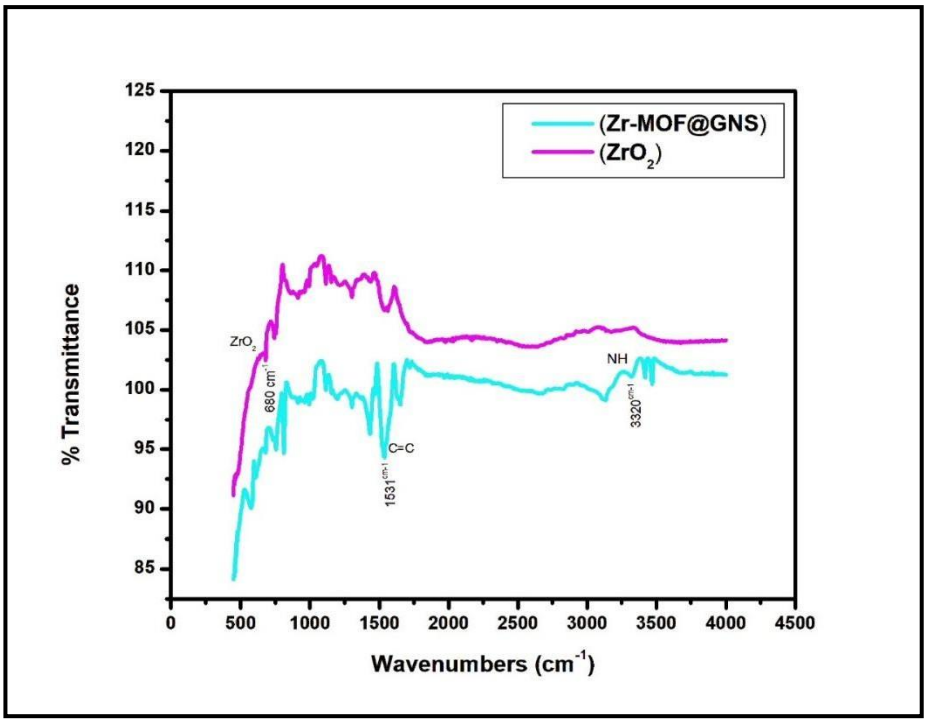

Fig1FT-IRspectralanalysisofZr-MOF@GNSandZrO_2

Fourier-Transform Infrared (FTIR) spectroscopy was employed to elucidate the chemical functional groups and confirm the successful synthesis of the Zr-MOF@GNSnanocomposite.ThespectraforthepristineZrO_2andtheZr-MOF@GNS composite are presented in Fig. 01

ThesynthesizedZrO_2 nanoparticlesexhibitedabroadabsorptionbandinthe rangeof400-700cm^{-1},which is characteristicofthestretchingvibrations of Zr–O bonds [21].Additional bands observed at 1630 cm^{-1} and 3400 cm^{-1} were assigned to the H–O–H bending and O–H stretching modes of water molecules,respectively,acommonfeatureinmetaloxideswithhighsurface area [22].
Theincorporationofmelaminewasevidencedbythedistinct N–Hstretching vibrations, manifesting as a series of medium-sharp bands in the 3200-3500 cm^{-1} region [23-24]. Furthermore, the C=C skeletal vibrations from the graphiticsp²carbon domains ofthegraphenenanosheets wereidentified as a shouldernear1500-1600cm^{-1},overlappingwiththecarboxylatesignals.The Zr–O stretching vibrations from the inorganic secondary building units (SBUs)oftheMOFcontributedtothebroadabsorptionfeaturesbelow800 cm^{-1} [25, 26].
The collective evidence from the FTIR analysis confirms the successful formationoftheZr-MOFstructure,itsfunctionalizationwithmelamine,and its composite nature with graphene nanosheets [27].

PXRDAnalysis

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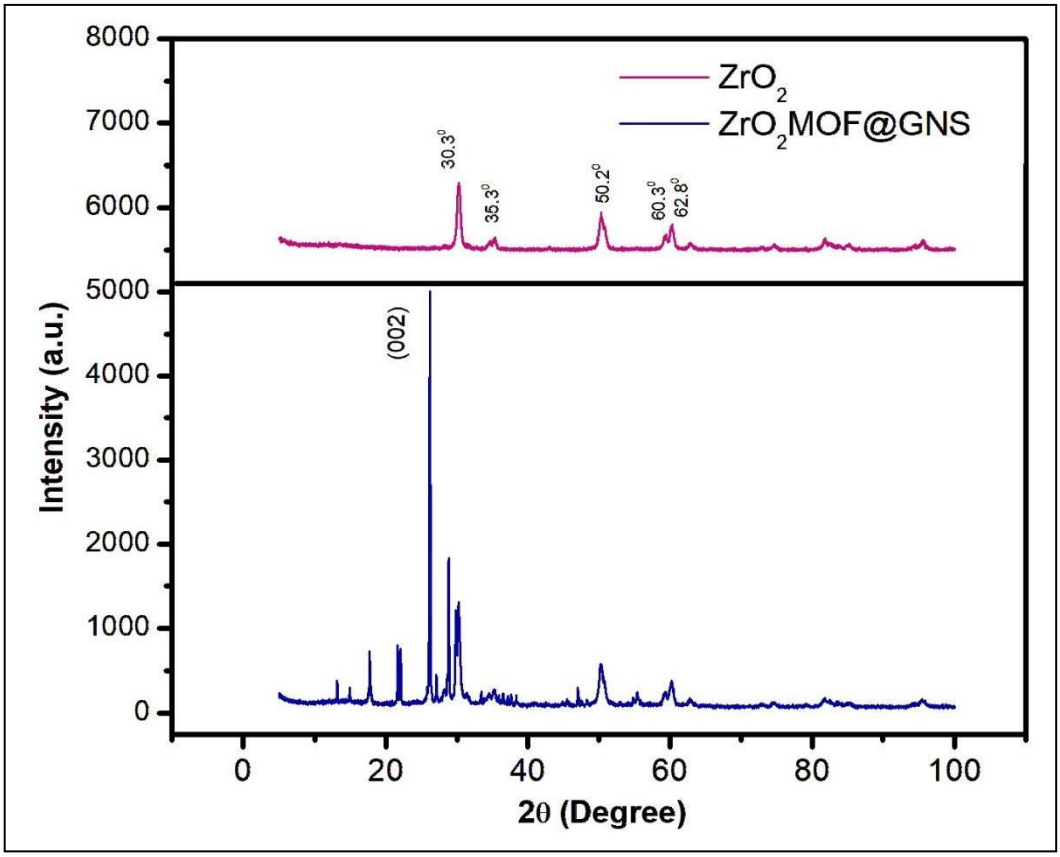

Fig3PXRDanalysisof(a)$ZrO_2$and(b)Zr-MOF@GNS

Major diffraction peaks appear at 2θ ≈ 30.3°, 35.3°, 50.2°, 60.3°, and 62.8°, consistentwiththe**tetragonalphaseofZrO_2(JCPDS49-1642)**.Thepresenceof sharp, well-defined peaks signals high crystallinity and phase purity, which directly correlates with the photocatalytic activity of ZrO_2-based systems.Such nanoscale,highlycrystallineZrO_2isacknowledgedforefficientchargeseparation during photocatalytic reactions a factor repeatedly demonstrated as enhancing pollutant degradation rates. Particle size calculation using the Scherrer equation

$$D = 0.9\,\lambda / \beta \cos\theta$$

yieldsanaveragesizearoundZrO_2is0.1101nmandZr-MOF@GNSis0.1022 nm as a result that closely matches values commonly reported for ZrO_2 nanostructures and GNS product formed.The subtle peak near 2θ ≈ 26.5° is attributed to the (002) plane of graphitic carbon, evidencing successful incorporation of graphene nanosheets within the composite. The absence or attenuationofsomeparentZrO_2peaksandtheemergenceofneworbroadened reflections in the composite confirm the formation of the zirconium MOF structure and integration with GNS. Zr-MOF composites regularly highlight MOF-related manifold reflections spanning 2θ ≈ 20–30°, further supporting successful framework formation. [28-35]

FESEMAnalysis

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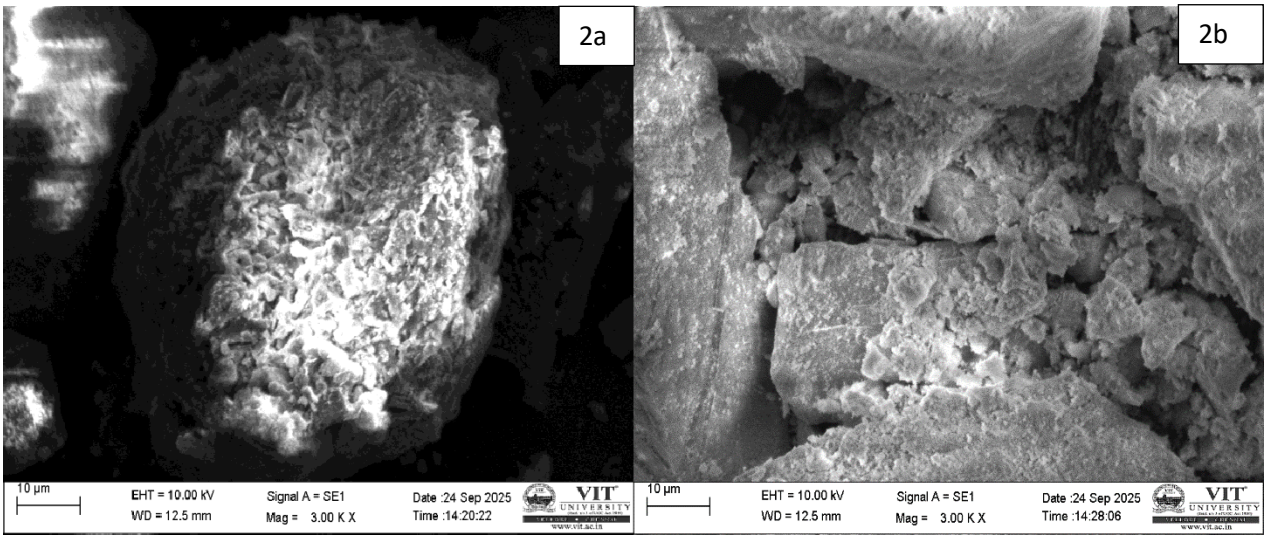

Fig2aand2bZrO_2SEM(2a),Zr-MOFSEM(2b) images

Thefig.2adepictsthattheZrO$_2$particlesareinamorphousstateDuetoaggregating oroverlappingofsmallerparticlestherearesomelargerparticles.TheSEMpictures clearlyexhibitthatthegrainsarerandomlydistributedwithsmallersizeanditis noticed that the particles are of homogeneous spherical shape, and are dusty and messy in with sharp edges in the corners and are slightly crystalline.Above images are information of the shape and structure of the particles synthesized [36]. Fig. 2b evident that the synthesized Zr-MOF@GNS is in crystalline state and are brittle in nature. The improved morphology is advantageous for photocatalytic and electrocatalyticapplicationasitensuresahighersurfaceareaandbetteraccessibility to active sites.

PhotocatalyticApplication

EffectofinitialconcentrationofAY99dye

InordertostudytheeffectofinitialconcentrationofSolutionbythe catalysts such as ZrO_2 and Zr-MOF@GNS, the amount of the Catalysts dosage is kept constant and different initial concentrations are varied in the particular time interval.

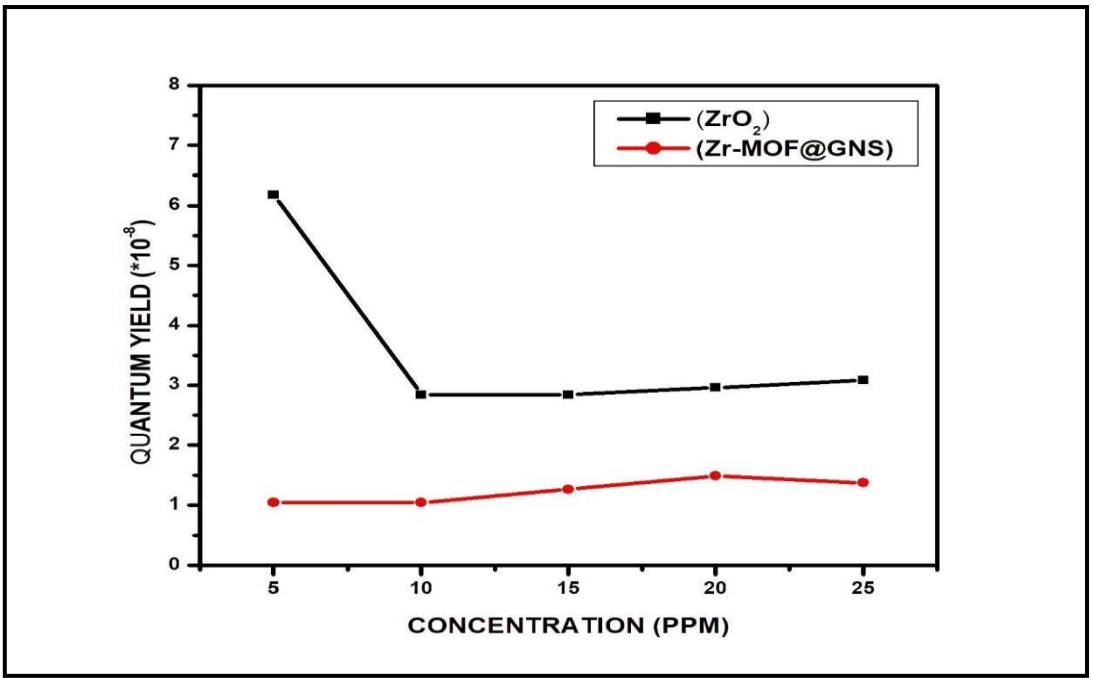

Fig4 - Effectofinitialconcentrationonthephotodegradationofay99undersolar light irradiation

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The observed results revealed that the quantum yield of the photocatalysis process increases with increase in initial concentration ofAY99 dye.This can be explained in terms of availability of active sites on the catalyst surface and the penetration of solar light into the suspension. The total active surface increases in the AY99 increases with increase in the concentration which tends to increase in the removal [37].

Thelengthoftheirradiationperiod

In order to study the length of irradiation period on the removal of AY99 dye by photocatalytic degradation process on $_{ZrO2}$ and Zr-MOF@GNS, the degradation experiments are carried out at constant amount of the catalysts and optimum initial concentration but varying the irradiation period.

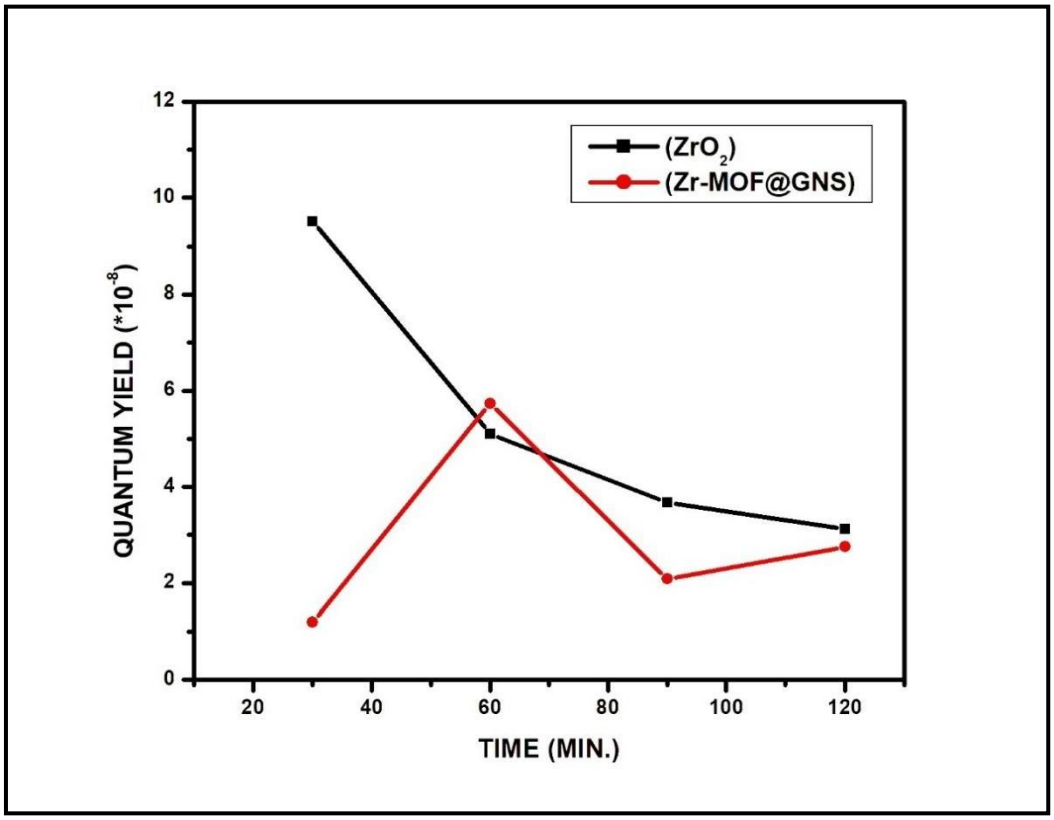

Fig5:Thelengthoftheirradiationperiodofthephotodegradationofay99 under solar light irradiation

The length of irradiation period plays an important role in degradation process of pollutants.Thelengthofirradiationperiod isvariedfrom30to180minTheresults reveals that the quantum yield of the reaction decreases with increase in irradiation period. It is observed that Zr-MOF@GNS / solar light system exhibits better photocatalytic efficiency than that of $_{ZrO2}$ / solar light system. [38]

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Thecatalystdose

To study the effect of the catalyst dose, the amount of $_{ZrO2}$ and Zr-MOF@GNS is varied, the initial concentration ofAY99 dye solution in each case is kept constant at optimum value.

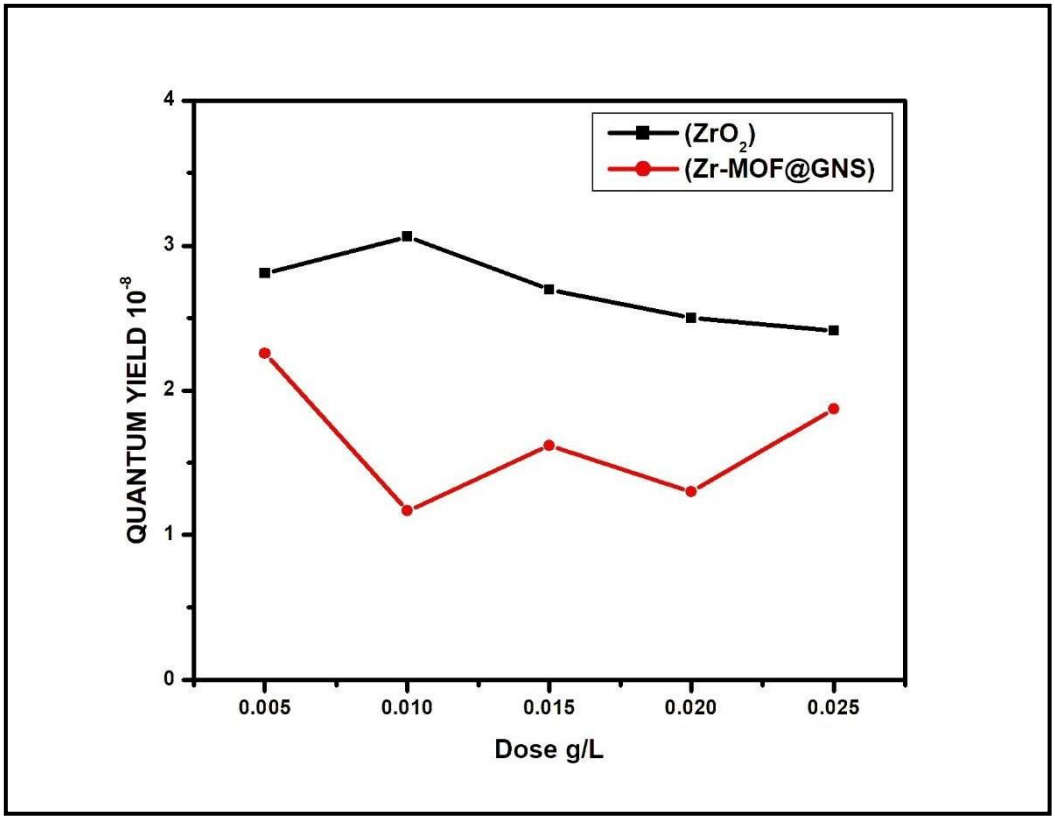

Fig6 - Thecatalystdosevariationofthephotodegradationof AY99undersolarlight irradiation

Optimizing the amount of $_{ZrO2}$and Zr-MOF@GNS is done in order to obtain the maximum amount of quantum yield in the photocatalytic degradation process. Henceinthisstudythequantityofthecatalyst isvariedfrom0.005g/Lto0.025g/L. It is noticed that the quantum yield of thereaction increases with an increase in the amountof$_{ZrO2}$andZr-MOF@GNS.Thisisduetothefactthatincreaseinthecatalyst dosethenumberofactivesitesonthesurfaceofthecatalystincreasesandhencethe quantum yield of the reaction increases.[39]

ThepHof thedye solution:

The photo degradation experiments are carried out at different initial pH at constant optimum initial concentration of AY99 dye solution, the amount of the catalyst and irradiation time.

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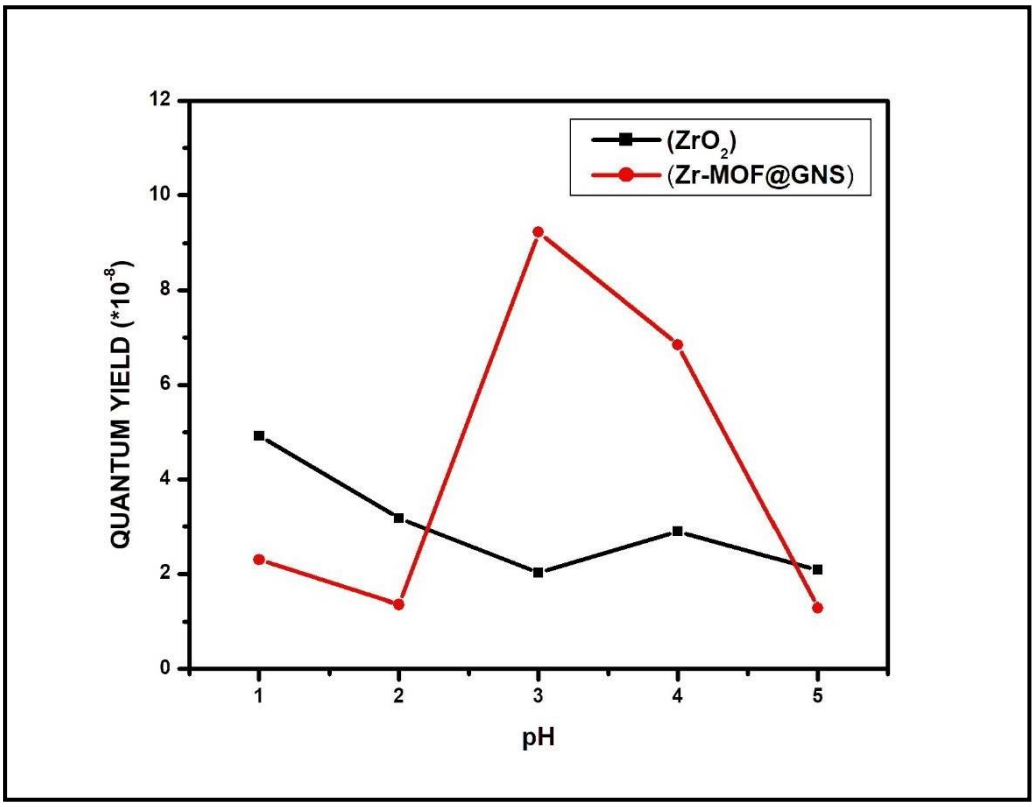

Fig7 - ThePhofthedyesolutionisvariedonthephotodegradationofay99under solar light irradiation

The pH value is one of the important factors influencing the quantum yield of the photodegradationprocessofpollutantespeciallydyes.TheAY99dyedegradation ishighlypHdependent.ThephotocatalyticdegradationofAY99dyeatdifferentpH valuesvaryingfrom1to5,clearlyshowsthatthequantumyieldefficiencyishigher inacidicmedium.TheZr-MOF@GNShavinghighefficiencyforphotodegradation of AY99 dye.[40]

4.0pictorialexplanation

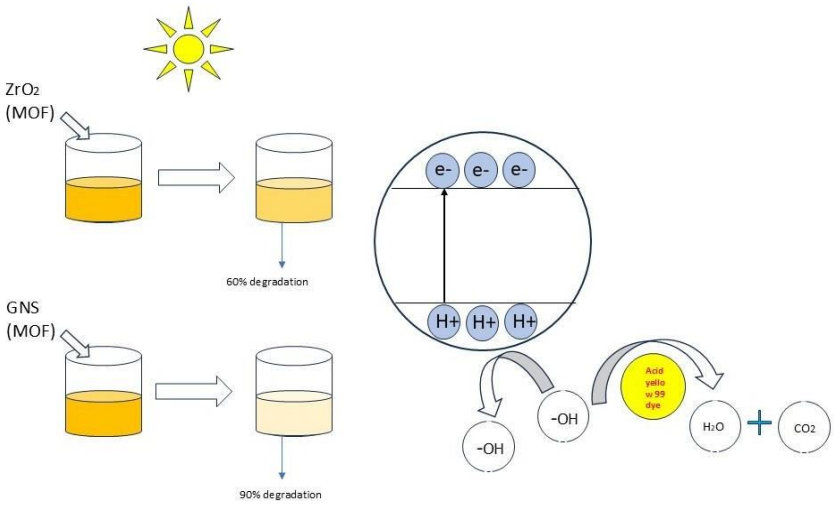

Fig9

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4.1MechanismofphotodegradationofAY99dye

Usingaphotocatalyst—usuallyasemiconductormateriallikeTiO_2,ZnOorZrO_2— to capture solar energy and break down dye molecules is known as photocatalytic degradation of hazardous dyes under sunlight. The photocatalyst creates electron-holepairsbyabsorbingphotonswhenexposedtosunshine.Reactiveoxygenspecies (ROS) like hydroxyl radicals (•OH) and superoxide anions (O_2^{-}•) are created when excited electrons (e^-) and holes (h^+) engage in redox processes. When the dye molecules are attacked by these very reactive species, they are broken down into smaller, less hazardous, or mineralized chemicals like CO_2 and H_2O. This environmentally friendly technique efficiently eliminates colors from wastewater.

Photonsfromsunlightwithenergyequaltoorhigherthanitsbandgapareabsorbed byzirconiumoxide.Positivelychargedholes(h^+)wereleftinthevalencebandafter these energised electrons (e^-) in the valence band were promoted to the conduction band.

Theelectron-holepairsinteractwithmoleculesintheirenvironment.Theholes(h^+) can oxidize water (H_2O) or hydroxide ions (OH^-) to generate highly reactive hydroxyl radicals (•OH). Simultaneously, the electrons (e^-) reduce oxygen molecules (O_2) to form superoxide anions (O_2^{-}•).

$$H^+ + H2O \rightarrow {}^{\bullet}OH + H^+ \quad (2)\ H^+ + OH^-$$

$$\rightarrow {}^{\bullet}OH \quad (3)$$

$$e^- + O2 \rightarrow O2^{-\bullet} \quad (4)$$

The dye molecules are attacked by the very reactive superoxide anions ($O_2^{-\bullet}$) and hydroxyl radicals (•OH). By cleaving bonds and starting oxidation processes, these radicals split the complex colour molecules into smaller pieces.

$$AY99 + \bullet OH/O2^{-}\bullet \rightarrow CO2 + H2O + otherharmlesscompounds \quad (5)$$

The dye eventually mineralizes into non-toxic substances including carbon dioxide (CO_2),water(H_2O)andinorganicionsasaresultoftheintermediatescreatedduring the breakdown process being further oxidized over time. The photocatalyst is not consumed during the reaction, is making the process sustainable. It can continue to catalyzereactions as long as it is exposed to sunlight andis not fouled ordegraded. Thisprocessiseco-friendlyandefficientfortreatingdye-contaminatedwastewater, as it utilizes sunlight (a renewable energy source) and does not produce harmful secondary pollutants. [41]

5.0APPLICATIONTOCONDUCTIVIY:

From the catalyst which are used in the photocatalytic degradationareverifiedinordertocheckttheconductivityofthesynthesized photocatalyst.

5.1ElectrochemicalreactionisstudiedusingZrO2andZr-MOF

FigdepictstheelectrochemicalacidbasetitrationreactionofNaOHvsHClusing $_{ZrO2}$and Zr-MOF@GNS as a electro catalyst. the observed results indicate that the end point of the reaction decreases significantly while using Zr-MOF@GNS compared to that of ZrO_2.

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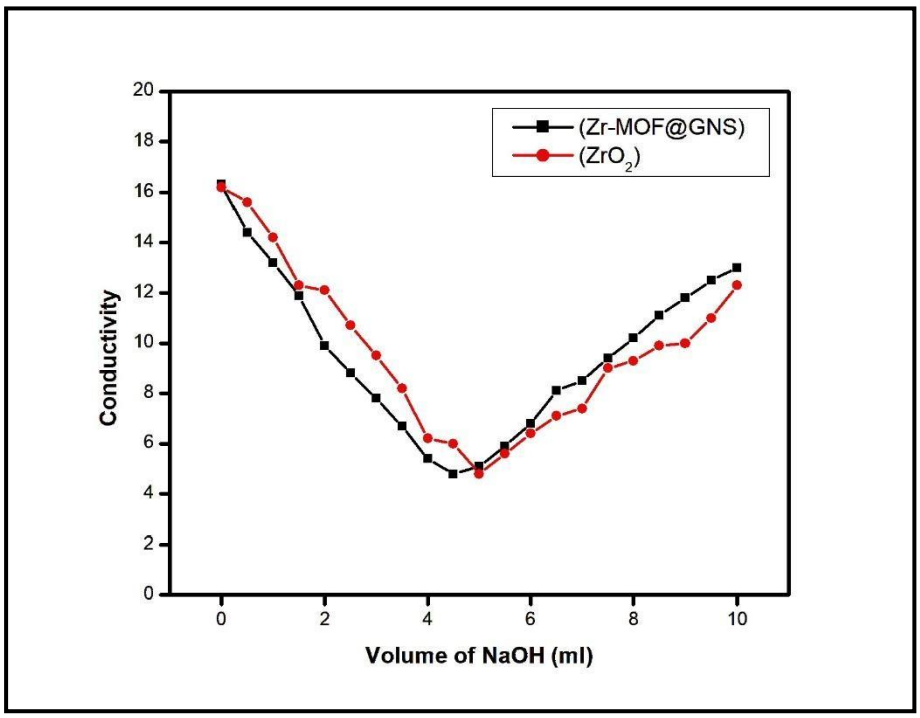

Fig8 ElectrochemicalstudiesofZrO$_2$andZr-MOF@GNS

6. CONCLUSION:

ThephotocatalyticactivityofZrO$_2$andZr-basedMOFisstudiedundersunlight as source for the photodegradation of AY99 dye. The effect of initial concentration,thelengthofirradiationtime,catalystdoseusedandpHofthedye solution is studied and the reaction conditions are optimized.The mechanismof photocatalyticdegradation takes place through the formation ofreactiveoxygen species(ROS)suchashydroxylradicals(•OH)andsuperoxideanions(O_2^{-}•).The electro chemical studies indicate that Zr-MOF@GNS increase the rate of the reaction.FromtheresultsitcanbeconcludedthatZr—MOF@GNScatalystand ZrO$_2$ act as photo and electro catalyst.

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