



Dye Degradation with suitable spinel processed by microwave assisted solution combustion synthesis

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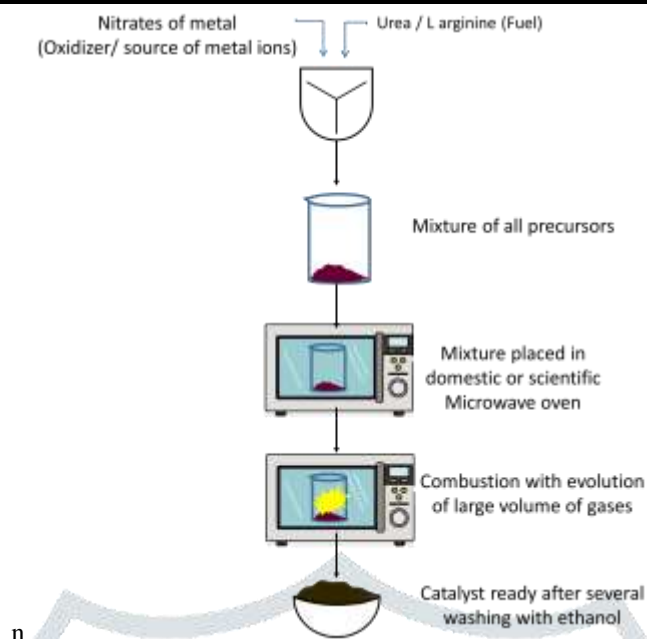
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Abstract: Dye waste water is the big concern from Industries after discharges high amount of water, it's effective from aquatic life to humans. Most parts of textile industrial dye impact to the environment like allergic reaction, respiratory, skin problems, irritation to mucous membrane and the upper respiratory tract have to the fore. In the textile sector, intoxicated dye chemicals are surrounded, it is problem for workers who do their job in this process. After the process, approximately 10 – 15 % dye is released into the environment. So, it is necessary to do the degrade dye and maintain TDS (Total Dissolved Solids) of water its necessary. In AOP (Advance oxidation process) gained attention in past decades, in photocatalyst is the simple activity to degrade dye by breaking rigid bond structure with suitable spinel. Tracking such parameters pH, irradiation time, initial dye concentration, contact time etc.

Ferrite spinel gained huge attention due to their wide application area from biomedical to wastewater treatment, pharmaceuticals, biomedical, electronic devices, and photocatalyst etc. The common ferrite spinel formula is $M\text{-Fe}_2\text{O}_4$ where M is Fe, Co, Mn, Zn, Cu, and Ni. Spinel Ferrite can be synthesized by conventional methods i.e., Sol-gel methods, Co-precipitation, Hydrothermal, which are slow and need more equipment facilities while the Microwave solution combustion (MSC) method is the promising method for the catalytic activity or any solid material. This method is fast, energy-efficient and needs lesser equipment than any others i.e., Sol-gel or Co-precipitation.

Herein microwave solution combustion method is reviewed in detail with insight into the effect of parameters. In this process, microwave irradiation is used to keep precursors at an excited state and make them highly reactive. Normally 2.45 GHz frequency microwave is used for this process. As this process is a solvent-free process. The most commonly used metal sources are nitrates of metals and fuel precursors are urea, L-arginine, glycerol etc. Urea is the most commonly used fuel because it gives less particle size. Different parameters such as fuel to oxidizer ratio, irradiation time, power of microwave and temperature and pressure can be optimized to get faster and better yield. This process does not need further calcination unlike sol-gel and co-precipitation but needs several items of washing with ethanol or n-butanol for purification of the final product. Finally, Characterize the sample by SEM, TEM, XRD, TGA, BET.

Though it is a novel process, in the end, it generates a large volume of gases produced during the combustion process. And this process can be used only for laboratory purposes till yet and some research is awaited.



Key words: Microwave Solution Combustion method, Ferrites, Spinels, Doped Spinels, dyes.

1. INTRODUCTION:

One of the major concerns from textile effluent dyes as waste product source produces carcinogenic effects into environment and human beings. Due to the huge wastage some allergic reactions occur into environment like skin irritation, eye problems, allergic diseases etc. Various types of Chemical Industries generated wastewater or pollutants such as textile and dye, food processing, iron and steel, Nuclear, pulp and paper industries. Dye waste water from industries produces 40-50 tons waste that is mix dye included Methylene blue, anthraquinone dye, Reactive blue 19, Rhodamine B, Reactive Blue 21, Methyl orange, Congo red, crystal violet, and many more. Our survey concluded that these dyes are attaining obstacles for an industry and life cycle. It also disturbed BOD and COD by toxicity in water. We all know that water is the natural source in our life. It is important for living. Highly toxic benzidine based dispersed dyes causes for aquatic life last year. Heavy metals complex dyes suffer the gills of the fishes. Further it is promising to degrade organic compounds to degrade dyes by the AOPs (Advance oxidation process). Researchers have generated interest overcome these details from dye effluents to protect our environment and health concerns. So, this issue motivated our work to ensure and collect all information on dye wastewater. Degradation strategies in terms of COD and reduce color without formation of by product.

Today's burning issue faced by waste water. The pollution of water resources by the dyes has become a major issue in environment problems. In textile dye effluent reactive dyes are the most problematic compound. Many industries discharges waste water which contain pollutant that are harmful. Removal of dyes from effluents before discharge into natural water bodies becomes environmental important.

There is a need to find alternative or modification in treatments which are effective in removing dyes from large volumes of effluents and are also effective. Various treatment or advance treatment methods are developed for the dyes degradation. Many processes for the wastewater treatment like chemical, physical and biological are used and these processes are not feasible. So advanced wastewater treatments are developed among which membrane technology as an advance separation technology has gained popularity and has become an important technology to treat and even reuse wastewater. Membrane processes such as microfiltration (MF), Ultrafiltration (UF), Nanofiltration (NF), and reverse osmosis (RO) have been adopted. In order to treat wastewater to improve the treatment efficiency and quality of water to meet the requirement of stringent legislations on water membrane treatment is widely used. Many conventional methods have been proposed to treat dye effluent which are very effective nor environmentally compatible and have shown to pose certain operational limitations and shortcomings. One of the method as best attentions for dye degradation

is that photocatalyst. It is promising technique with high efficiency and low cost in visible light. Photocatalyst is the greatest interest has developed. Solar energy active photocatalyst has been a promising material for an environment purification processes. Some efforts have been made to synthesize materials capable of utilizing solar spectrum for the photo degradation of industrial waste pollutants and dyes. The modification methods of photocatalyst involve noble metal deposition, transition metal ions doping, couple semiconductor etc. Photocatalyst utilize light energy to carry out oxidation and reduction reactions. When irradiated with light energy, an electron is excited from the valence band to the conduction band of the photocatalyst leaving a photo generated holes (h^+).

Semiconductors are widely used for photocatalysts process. In semiconductors like TiO_2 , ZnO , Fe_2O_3 , CdS , GaP , ZnS . And multi component oxides as the catalyst to degrade azo dye by attractive attention. But some kind of demerits by processing because it works only in Ultra violet source, requires high amount of dosage, separation problem and extensive sludge disposal problems. Several studies shows that ZnO is nearly equal to TiO_2 because it generates H_2O_2 efficiency to degrade dyes. On the other hand spinel catalyst with magnetic property has been investigated to overcome the drawback of semiconductor. Generally, we known spinel forms are AB_2O_4 – Transition metal cation and theses novelty of catalyst shows better magnetic and electrical properties.

Class	Characteristics	Fibre	Method of application
Acid	Anionic water soluble	Nylon, wool, silk	Neutral to acidic dry bath
Basic	Cationic water soluble	Modified nylon, polyester	Acidic dye bath
Direct	Anionic water soluble	Cotton, rayon, nylon, leather	Neutral or alkaline bath containing
Disperse	Very low water soluble	Polyester, polyamide, acetate, plastic, plastic acrylic.	High temperature pressure
Sulphur	Colloidal, insoluble	Cotton, rayon	Aromatic substrate vetted sodium sulphate or re-oxidized to insoluble sulfur containing products on fire.
Vat	Colloidal or insoluble	Cotton, rayon	Water insoluble dye solubilized by reducing with sodium hydro-sulphite on fibre or re-oxidised.

Table1: Classification of Different dyes from textile industries and its application

1.1 Industrial limits for waste water:

The large quantity of dye produced in the world and discharge to the environment are not available, so estimated production rates is 10,000 tons/ year and losses around 1 to 2 % during production. Environmental problems is huge concern generated by central pollution control board in prevention act. Approximately 1.1 billion people drinking unsafe water and this issue shows by government or by scientists. The formation in 1974 of ecological and toxicological association of dyestuffs manufacturing industry, aims to minimize environment damage, protect users, consumers and to cooperate fully with government and public concerns over

impacts of products. Thus textile in India release maximum wastewater and these textile are in Surat, Ahmedabad, Mumbai, Kanpur, Coimbatore. Due to the increase of wastewater discharge increasingly stringent legislation on priority the search for the appropriate technologies. Even below 1 ppm of dye in waste can be visible than organic substances so it is more important to remove from environment.

Characteristics of wastewater of textile	Values
pH	9.8-11.8
Total alkalinity	17-22 mg/l
BOD	760 – 900 mg/l
COD	1400- 1700 mg/l
TDS	6000-7000 mg/l
Total chromium	10-13 mg/l

Table 2.2. Composition of cotton textile mill waste (Khatri et al., 2015).

In order to maintain the conditions under water act the applicant shall provide adequate effluent treatment system in order to achieve the quality of the treated wastewater as per the Gujarat pollutions control board norms.

Chemical	Quantity kg/month
Acetic acid	1611
Ammonium sulphate	858
P V Acetate	954
Wetting Agent	125
Caustic soda	6212
Softener	856
Organic solvent	247
Formic acid	1227
Soap	154
Hydrosulphites	6563
Hydrochloric acid	309
Hydrogen Peroxide	1038
Leveling and Dispersing agent	547
Solvent 1425	321
Oxalic acid	471
Polyesthylene Emulsion	1174
Sulphuric acid	678
Disperse dyes	1500
Vat dyes	900
Sulphur dyes	300
Reactive dyes	45

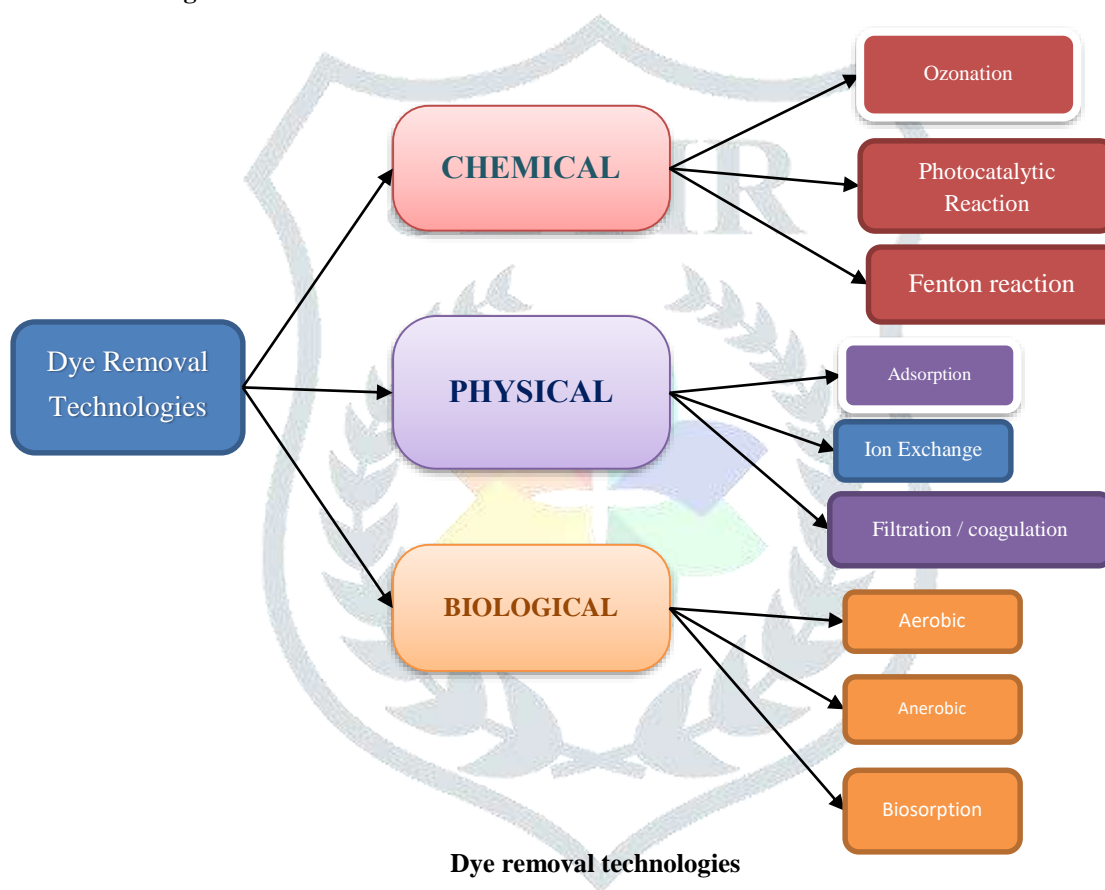
Table 3: Major chemical dyes and synthetic textile mills (Aggadi, 2020)

Azo, anthraquinone, triarylmethane and phthalocyanine dyes are the most important dye groups. Phthalocyanines in the presence of a metallic reaction of dicyanobenzene in the presence of a metallic type of Cu, Ni, Co, Pt. Copper phthalocyanines are the main

derivative of this dye class, due to their beautiful blue to green shades, high dyeing resistance, excellent chemical stability, light fastness and resistant to bacterial stability, light fastness and resistant to bacterial degradation under aerobic or anerobic conditions. (Aggadi, 2020).

Many preparation techniques or method are sol gel, co-precipitation, solid -state, hydrothermal, microwave solution combustion. But some conventional method limitations high heating rate, emitting poisonous gases, long time requirement, higher gain size, time consumptions etc. But Microwave solution combustion overcome limitations, its effective and energy efficient and time saving method.

1.2 Dye removal technologies



Generally, dye degradation techniques majorly explored in physical, chemical and biological processes. Adsorption, irradiation, and ion exchange comes under physical methods which are out of practice. Ozonation, advance oxidation processes fentons process are classified on chemical methods which are in use currently. Biological methods like aerobic degradation, anaerobic degradation, fungi and algae are also in practice. Considering all these methods AOPs is concluded to the best because its efficiency on degradation due molecules. AOP process has several methods like photosynthesis, catalysis, sonolysis etc. Photocatalysis exhibit almost complete degradation of dyes in wastewater.

2. METHODS FOR CATALYST PREPARATION:

2.1 Microwave solution combustion Method:

2.1.1 Materials:

Commercially reagents catalyst preparation with AR, LR, ACS grade chemicals can be used. All the reagents were analytical grade obtained from Merck Co. and were used as received without further purification.

Name of Oxidizers	Chemical formula	Name of Fuels	Chemical formula	Solvents
Nickle nitrate hexahydrate	$\text{Ni}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	Glycine	$\text{C}_2\text{H}_5\text{NO}_2$	Benzene (C_6H_6)
Iron nitrate nonahydrate	$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	Sucrose	$\text{C}_{12}\text{H}_{22}\text{O}_{11}$	Ethanol ($\text{C}_2\text{H}_5\text{OH}$)
Magnesium nitrate hexahydrate	$\text{Mg}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	Urea	$\text{CH}_4\text{N}_2\text{O}$	Methanol (CH_4O)
Copper (II) nitrate	$\text{Cu}(\text{NO}_3)_2 \cdot 3\text{H}_2\text{O}$	Cetyltrimethyl ammonium bromide (CTAB)	$\text{C}_{19}\text{H}_{42}\text{BrN}$	2-methoxyethanol ($\text{C}_3\text{H}_8\text{O}_2$)
Cobalt nitrate	$\text{Co}(\text{NO}_3)_2 \cdot 6\text{H}_2\text{O}$	Citric acid	$\text{C}_6\text{H}_8\text{O}_7$	formaldehyde (CH_2O)
Cobalt acetate	$\text{Co}(\text{CH}_3\text{COO})_2 \cdot 4\text{H}_2\text{O}$	Sorbitol	$\text{C}_6\text{H}_{14}\text{O}_6$	Water (H_2O)
Manganese nitrate	$\text{Mn}(\text{NO}_3)_2 \cdot 4\text{H}_2\text{O}$	Ethylenediaminetetraacetic acid	$\text{C}_{10}\text{H}_{16}\text{N}_2\text{O}_8$	Kerosene
Nitric acid	HNO_3	Ethylene glycol	$\text{C}_2\text{H}_6\text{O}_2$	Acetone (cleaning)
Ammonium nitrate	$\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$	Oxalyl Dihydrazide	$\text{C}_2\text{H}_6\text{N}_4\text{O}_2$	
		L-arginine	$\text{C}_6\text{H}_{14}\text{N}_4\text{O}_2$	

Table 4: All oxidizers and fuels for propellant

2.1.2 Procedure for an Experiments:

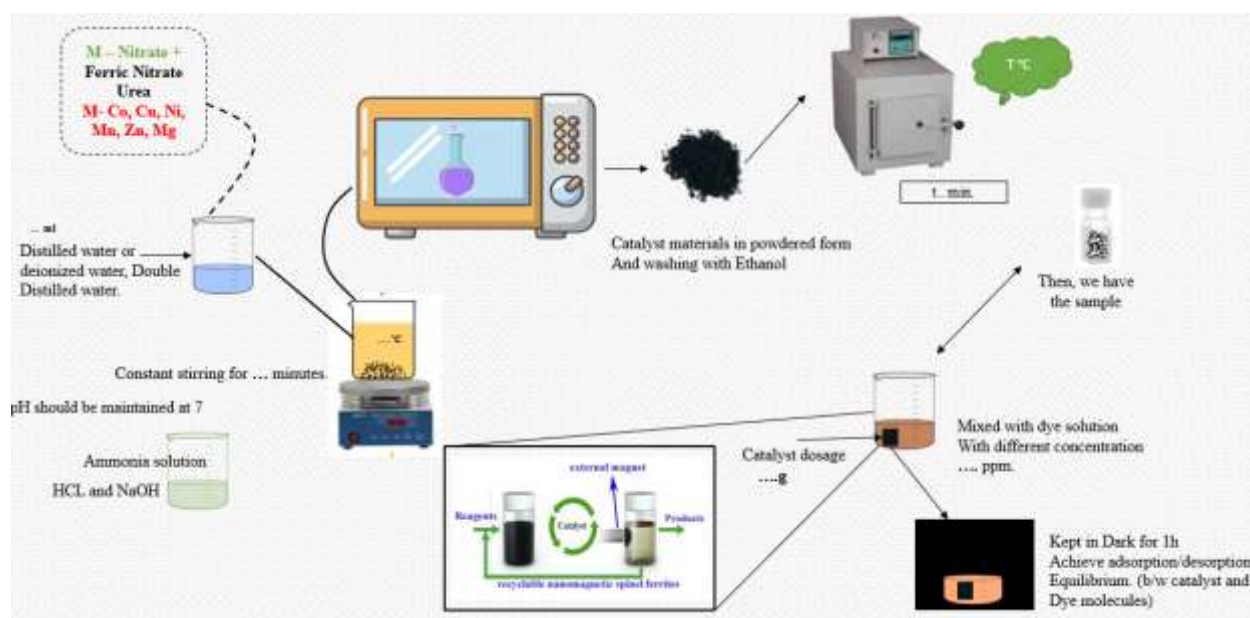


Figure 1: Microwave solution combustion method with dye degradation

Required amounts of iron nitrate $\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, mixture with divalent metal cation with fuel consisted reducing agent and it also capable for combustion reaction. The amount of mixture calculated by stoichiometry relation listed in Table 4 above. Then proceed to make propellant dissolved in distilled water for solubilizing. Only for mixture of fuels, pH of solution precursor was adjusted to 7 with addition of ammonia solution (NH_4OH) under continuous stirring. The mixture slowly evaporated at 80°C until a viscous solution was formed. The mixture poured into a round bottom flask and heated until it transformed into a gel; by further heating up to 250°C , ignition reaction started from a point and propagated spontaneously. The combustion gases were bubbled in a large beaker filled with water. The obtained powders by hand-crushed with a pestle.

2.1.3 Photocatalysts activity

Photocatalyst are recently recognized as promised products for dye degradation due to various uses, simple operating also operates in moderate environments without due to various uses, simple operating also operates in miderate environments without special equipment. In dye degradation under visible light as source and the metal ferrites in efficiency for catalytic degradation. In visible light illumination excited electrons from valence band to the conduction band and the left holes at Valance band to react the surface adsorbed by water molecules to form highly reactive hydroxyl radicals OH^- and H^+ ions. Conversely dissolved oxygen adsorbs electron from the conduction band and produce superoxide anion radical oxygen. Both hydroxyl and superoxide radical will degrade dye molecules. So, this is the mechanism for dye degradation here is the figure demonstrate the phenomena.

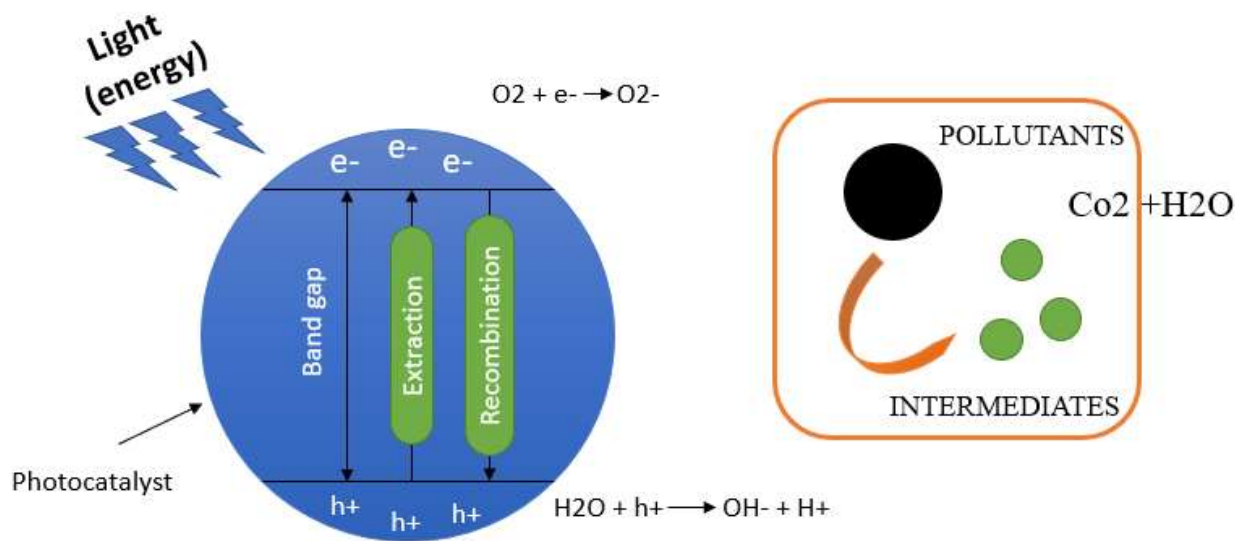


Figure 2: Photocatalytic degradation

Effect of surface area on the degradation of dye:

Material with a high specific area more effective for dye degrade by photocatalytic activity. When surface area of synthesis particle increases the particle size decreases, where as the electron hole pair enhances rate. If higher surface absorbed of the sample, show good photocatalytic property.

Effect of different times:

Effect of hydrogen peroxide as oxidant: the rate of degradation increases increase dye to the decomposition of H₂O₂ is the creation of OH⁻ radical by e⁻/h⁺ pair recombination on the surface of the sample. The electron created reaction are directly reacted by Fe³⁺ with H₂O₂ to form OH.

CHARACTERIZATION:

FTIR measurement: Degraded dye products were collected after color removal reaction by centrifugation and vacuum dried. The IR spectra of dye and degraded dye product after the decolorization procedure can be record in FTIR: 4000 – 400 cm⁻¹ range. FTIR spectra give a reasonable hint of the modifications happening in a dye molecule because of decolorization as a result of the vanishing of existing peaks. Thus, the degradation of dye by NPs included the simultaneous adsorption and catalytic of dye occurring on NPs.

XRD: Structural configuration, crystallize size, space group. The crystallite size calculated employing Scherrer formula. If the crystalline size is decreases also enhances the large surface area. The prepared sample was formed with cubic phase with face-centered lattice and it shows. Fd3m space group. Study defines the intensity of the peaks differs which exhibits the tough crystallization because of the large radii of it using rare earth metals which has been used for doping. XRD patterns confirm the formation of single phase spinel catalyst without impurities. The average crystallite size (D) was calculated using Debye Scherrer's formula.

The breadth of the bragg peak is the combination of both instrument and sample broadening effects. Hence, the line broadening dye to the instrument has to be decoupled with that of the sample by recording the diffraction pattern and studying the line broadening of a standard material such as silicon. The instrument corrected broadening, b corresponding to the diffraction peak of zinc aluminates was estimated using relation.

$$D = \frac{0.9 \lambda}{\beta \cos \theta}$$

The effective particle size (D) and the strain component were also evaluated using Williamson – Hall method. If the slope shows negativity it means the compressive strain, whereas the appearance of a positive slope indicates the possibility of tensile strain. In

the present study, a positive slope for all the samples reveals the presence of tensile strain in the composites. The X-ray density (ρ_x) for each sample was calculated by the relation.

$$\rho_x = ZM / NV$$

where, Z is the number of molecules per unit cell of spinel lattice (Z=8), M is the molecular weight, V is the unit cell volume, and N the Avogadro's number.

The percentage porosity (P) of the spinel was calculated by using the formula

$$P = 1 - [\text{Bulk density} / \text{X ray density}] * 100$$

The reduce in bandgap energy will make the catalyst capable of having efficiency in visible light which is present largely in solar light and can give good photocatalytic efficiency.

The vibrational, rotational and lattice defects present on the samples were clearly found by Raman analysis.

Scanning electron microscopy:

Morphological, development of clear nanoparticles with reduce in size is also evident from the image. The particles at 500 nm scale shows better grown nanoparticles with less agglomeration on the surface of the grown nanoparticles. While doping agglomeration completely reduced the nanoparticles grown and more clear surfaces. At 500 nm scale, the image will clear support.

EDAX: It confirms the elemental configuration.

Thermal gravimetric analysis (TGA): It is widely used to investigate the thermal decomposition of NPs to determine the thermal decomposition kinetic parameter. These parameters can be used to obtain a better understanding of thermal stability of catalyst.

Magnetization: The magnetization behavior of catalyst or doped catalyst can investigation with VSM (vibrational sample magnetometry) by seeping the external magnetic field. Magnetization plotted against, applied field (H) behavior plots. The values of coercivity and retentivity obtained from the M-H curves, the low values are attributed to the characteristic of magnetic NPs where thermal fluctuations are sufficient to overcome the anisotropy energy barrier, thus allowing the magnetization to spontaneously reverse the direction, Upon increasing the amount of doping as tends to super magnetization behavior, However increase in the doping concentration by a decrease in the coercivity value decreased due to the thermal fluctuations can be, it assisting the overcoming of anisotropy energy barrier. The smaller values of squareness ration M_r / M_s less than 0.5, according to stoner – wolfarth indicate the nanoparticles process uniaxial anisotropy. The squareness ratio classifies the materials to having cubic anisotropy. The magneto crystalline anisotropy constant (K) of the NPs obeying uniaxial anisotropy is calculate using Browns relation.

$$K_1 = \frac{H_c M_s}{0.985}$$

It has been observed that anisotropy constant (k_1) decreases at higher calculations temperature ($^{\circ}\text{C}$).

Uv visible spectroscopy: It shown absorbance as part of visible region 500-780 nm. The performance should be attributed to the efficiency transfer of photosensitivity from highly conjugate organic linker. The optical band gaps found by Tauc plot. Generally the bang gaps play a important role in determining their photocatalyst degradation efficiencies towards the organic pollutant decontamination. Narrow band gaps towards more conductive to the visible light absorption of catalysts to generate charge separated excited states, which is crucial state to determining the efficiency chemical energy from solar energy. The E_g (band gap) values determine by Kubleka munk equation $F(R) = \alpha = (1-R)^2 / 2 R$ and transformed formula $[F(R)h\nu]^2 = A (h\nu - E_g)$.

Where,

R = Reflected light

α = optical absorption coefficient

$h\nu$ = energy of incident photon

E_g = Band gap energy

A = transition probability rate constants

The band gap energy constructed in Tauc plot of Kubelka–Munk function $[F(R)h\nu]^2$ (ordinate) versus $h\nu$ (abscissa) (Qing Li a, 2018) and precisely determined as the abscissa values of intersection between the tangent line of energy axis and the line extrapolated from the linear portion of the adsorption edge (Qing Li a, 2018).

CONCLUSION:

Microwave solution combustion or solution combustion method both works collimated each other. The great propellent discovery found in microwave and easy reaction with less contact time to product suitable catalyst for dye degradation. Our study shown microwave comes drawback in large number of gases produce during exothermic reaction with spinel ferrites but its produce good magnetization for easy separation with dye. Solution combustion assisted work the catalyst for reduce particle size with certain amount to temperature life in muffle furnace during calcination and produce crucial separation. Saturation magnetism is good results shown by our study. So the microwave is very effective work for dye degradation as application.

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