

# Microwave Solution Combustion Method for Synthesis of Spinel Ferrite and their Magnetic Properties, Reusability, Particle Size, Fuels, Surface Area, Dye Degradation Application

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## Review Article

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## ABSTRACT

Ferrite spinel gained huge attention due to its wide application area from biomedical to wastewater treatment, pharmaceuticals, biomedical, electronic devices, photocatalyst etc. The common ferrite spinel formula is  $M-Fe_2O_4$  where M is Fe, Co, Mn, Zn, Cu, and Ni. The Microwave solution combustion (MSC) method and Solution Combustion Synthesis (SCS) is promising methods for catalytic activity or any solid material. This method is fast, energy-efficient and needs lesser equipment than any others. MSC 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 but needs several items of washing with ethanol or n-butanol for purification of the final product. 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 on rare earth metals doped with spinel. But in SCS, directly jump to calcination after precursor homogenized at neutral pH i.e., 7. Both Methods can work collimated if required a higher degree of separation with better yield and magnetic separation susceptibility during implementation on degradation on dye.

## INTRODUCTION

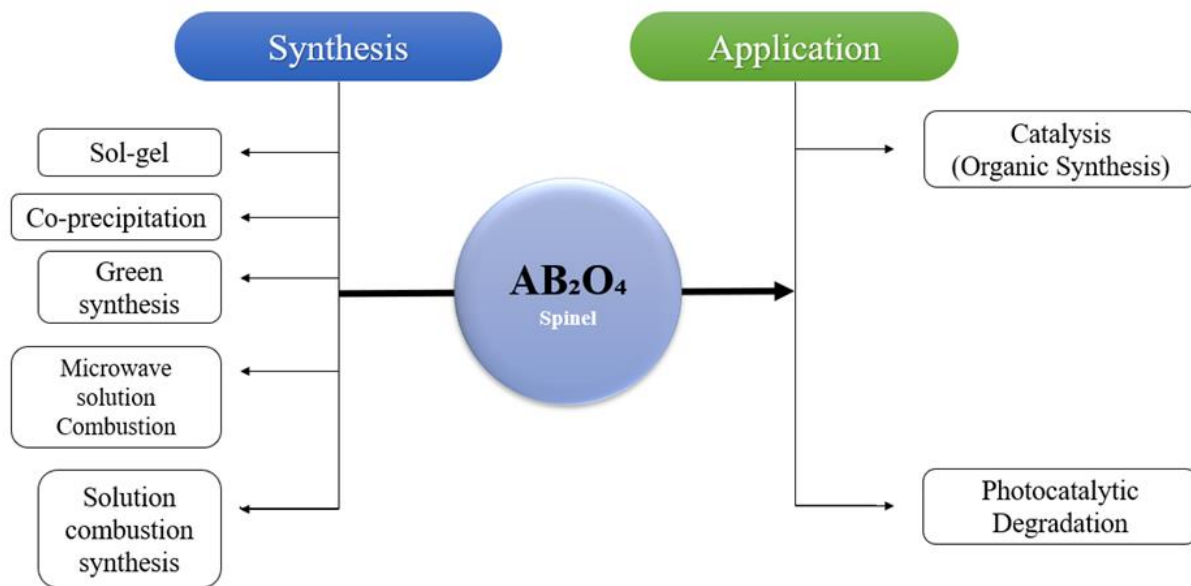
Spinel can be synthesis by sol gel, co-precipitation, micro emulsion, solid state, Hammer's method, microwave combustion method, sol gel auto combustion method, solution combustion method etc. Microwave solution method overcome from other methods because it's efficient, time saving, fast route for preparing spinel ferrites and doping of spinel ferrite also going to new generation catalyst for dye degradation as application. Spinel ferrites due their outstanding magnetic and electrical properties or high chemical and thermal stabilities. Generally, spinel ferrite expressed as  $MFe_2O_4$ . Where M is known as Transition metals [1-5]. It is kept voids between in Tetrahedral and Octahedral containing A and B sites cations in spinel ferrite structure. Here, in Inverse spinel kept Iron (III) trivalent cation fixed and other divalent cation can be arranged as per requirement. It is widely reported that magnetic, electrical and chemical properties of nanocrystalline ferrites are very sensitive to processing techniques. The inversion parameter and magnetic are strongly dependent upon size, shape, stoichiometry, crystalline, and surface groups all which are also controlled by preparation and post preparation method [6,7].

## LITERATURE REVIEW

Solution combustion synthesis also synthesized and increasingly production of ferrites due to its magnetic properties and possibility of producing highly pure and homogenous structure powered and low cost, short times. SCS is also called other route to processed ferrites. It consists of combination of both oxidizer and fuel ration for combustion in aqueous medium. The mixture is heat up until it reaches self-sustaining ignition is fast and highly exothermic combustion. The precursor solution is then combusted by extra heating up to the mean temperature (200-500 °C) to form the final product directly. Sometimes, it is required to calcine the combusted products at higher temperature (<700 °C). The catalyst powder is strongly dependent upon the F/O ratio. The F/O ration maintained is equal to 1. The powder amounts are crucial factors for turning the phase and micro structured of combusted powders [8-10].

Here, I this paper reviewed both on Microwave solution combustion synthesis and Solution Combustion Synthesis (SCS) method. Compare on both methods for their external routes (Figure 1).

**Figure 1.** Microwave solution combustion synthesis and Solution Combustion Synthesis (SCS) method. Compare on both methods for their external routes.



Spinel ferrite can be utilized for dye degrade from waste water. The dyeing and textile industry is responsible for dye discharge as well as plethora or other hazardous substance (either inorganic or organic). The dye waste water causes several effects into environment [11-13]. It causes asthma problems, carcinogenic diseases, skin irritation, eye sight problems, and many others. The problem of dye contaminated water is especially evident in Asia, which contributes about 50% of textile exports and more than 50% of world’s consumption of dyes [14,15]. It is clear that only discharged water need to degrade. The quality of waste water already announced as per CBCP (Central pollution control board) and GCPC (Gujarat pollution control board) [16-18].

The majority of industrial dyes and impacted into environment such dyes are Azo dyes, Reactive dyes, Anthraquinone dyes, Acidic and basic dyes [19,20]. Azo make up over 60% of the synthetic dyes used industrially and followed by other dyes. Azo (Reactive dyes) are easily available in cheap cost. Its stability in water is high. Dye the chromophore group (-N=N-) linkages. And sulphonic group present in (SO<sup>3-</sup>) in reactive dyes. The types of reactive dye are in table given below (Table1) [21-23].

**Table 1.** Categories of reactive dyes.

Categories of reactive dyes
Reactive turquoise blue H5G
Reactive navy blue HE2R
Reactive blue 171
Reactive turquoise blue MGN
Reactive red brown
Reactive yellow
Reactive purple
Congo red reactive dyes
Reactive orange dyes etc.

MATERIALS AND METHODS

Microwave solution combustion method

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 (Table 2).

Table 2. All oxidizers and fuels for propellant (SCS and MSCM) methods.

Name of oxidizers	Chemical formula	Name of fuels	Chemical formula	Solvents
Nickle nitrate hexahydrate	Ni (NO <sub>3</sub> ) <sub>2</sub> · 6H <sub>2</sub> O	Glycine	C <sub>2</sub> H <sub>5</sub> NO <sub>2</sub>	Benzene
				(C <sub>6</sub> H <sub>6</sub> )
Iron nitrate nonahydrate	Fe (NO <sub>3</sub> ) <sub>3</sub> · 9H <sub>2</sub> O	Sucrose	C <sub>12</sub> H <sub>22</sub> O <sub>11</sub>	Ethanol
				(C <sub>2</sub> H <sub>5</sub> OH)
Magnesium nitrate hexahydrate	Mg (NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O	Urea	CH <sub>4</sub> N <sub>2</sub> O	Methanol (CH <sub>4</sub> O)
Copper (II) nitrate	Cu (NO <sub>3</sub> ) <sub>2</sub> ·3H <sub>2</sub> O	Cetyl trimethyl Ammonium Bromide (CTAB)	C <sub>19</sub> H <sub>42</sub> BrN	2-methoxyethanol (C <sub>3</sub> H <sub>8</sub> O <sub>2</sub> )
Cobalt nitrate	Co (NO <sub>3</sub> ) <sub>2</sub> ·6H <sub>2</sub> O	Citric acid	C <sub>6</sub> H <sub>8</sub> O <sub>7</sub>	formaldehyde (CH <sub>2</sub> O)
Cobalt acetate	Co (CH <sub>3</sub> COO) <sub>2</sub> ·4H <sub>2</sub> O	Sorbitol	C <sub>6</sub> H <sub>14</sub> O <sub>6</sub>	Water (H <sub>2</sub> O)
Manganese nitrate	Mn (NO <sub>3</sub> ) <sub>2</sub> ·4H <sub>2</sub> O	Ethylenediamine tetraacetic acid	C <sub>10</sub> H <sub>16</sub> N <sub>2</sub> O <sub>8</sub>	Kerosene
Nitric acid	HNO <sub>3</sub>	Ethylene glycol	C <sub>2</sub> H <sub>6</sub> O <sub>2</sub>	Acetone (cleaning)

<b>Ammonium nitrate</b>	$Al(NO_3)_3 \cdot 9H_2O$	Oxalyl Dihydrazide	$C_2H_6N_4O_2$	
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## RESULTS AND DISCUSSION

### Required equipments

#### Microwave chemistry

- Inside the strong metal box, there is a microwave generator called a magnetron. When you start cooking the magnetron takes electricity from the power outlet and converts it into high-powered, 12 cm (4.7 in) radio waves.
- The magnetron blasts these waves into the food compartment through a channel called a wave guide.
- The food sits on a turntable, spinning slowly round so the microwaves cook it evenly.
- The microwaves bound back and forth off the reflective metal walls of the food compartments, just like light bounces they not simply bound off. Just as radio waves can pass straight through the walls of your house, so microwaves penetrate inside the food. As they travel through it, they make the molecule inside it vibrate more quickly.
- Vibrating molecules have heat so, the faster the molecules, the hotter the food becomes. Thus, the microwaves pass their energy onto the molecules in the food, rapidly heating it up.

The only loss factor in microwave is energy loss in a dielectric material due to slow polarization or some other dissipative phenomenon.

#### Muffle furnace

It is the equipment for heating or combustion of the material significantly in high temperature while keeping it contained chemicals or other substances. It is usually lined with stainless steel, making them largely corrosive. Different sample heated in furnace around 400°C-2000°C. Depend on the size of the muffle and various requirements in heating elements (Kanthal resistive wire, Silicon carbide rods, Molybdenum Disilicate) for higher temperature around <1500°C. PID based controller arranged in muffle furnace. The controller set out by set value and Process Value (PV). High density ceramic fiber blanket is used as insulation to keep the outer surface at minimum temperature (Table 3 and Figure 2).

**Table 3.** Basic specification of muffle furnace.

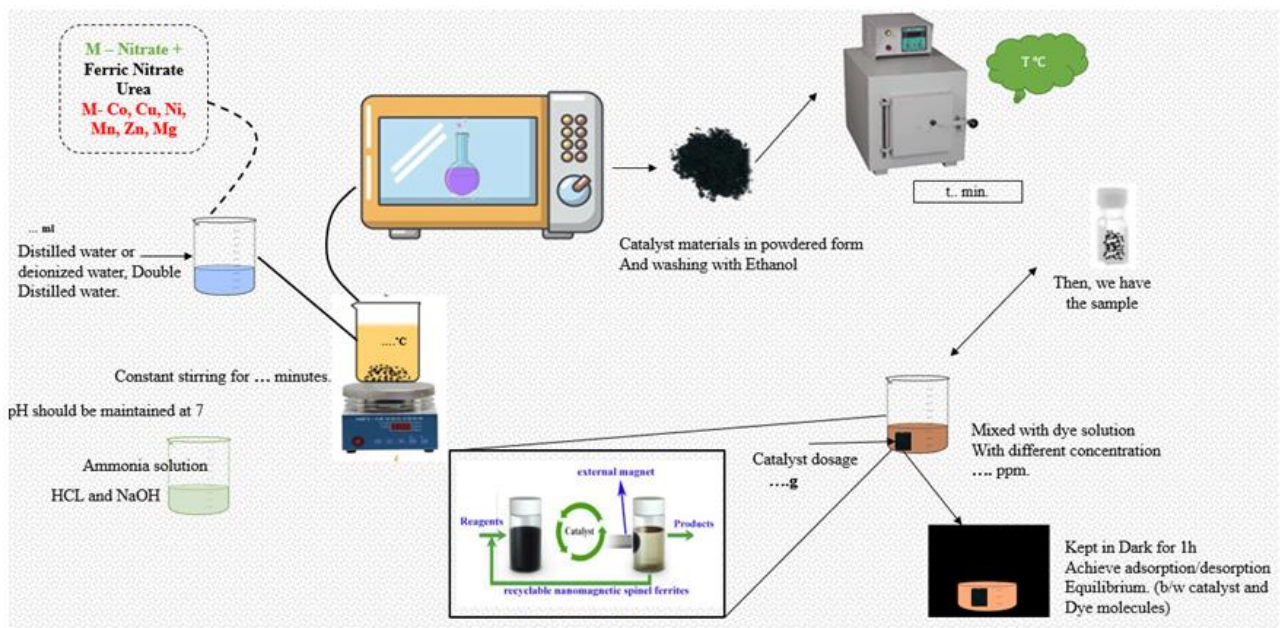
<b>Max temperature</b>	<b>1100°C</b>	<b>1400°C</b>
Continuous operating temp.	1000°C	1300°C
Heating element	Kanthal wire	Silicon Carbide (SiC) rods
Thermocouple	K Type	R Type
Chamber MOC	Ceramic fiber board	Ceramic zirconium board
Exterior MOC	Mild steel powder coated	Mild steel powder coated
Insulation	Ceramic fiber blanket	Ceramic fiber blanket
Temperature controller	PID controller with SCR power control	PID controller with SCR power control
Power supply	220 Volts 50Hz	3 Phase 440 volts 50Hz (with energy saving transformer)

	assembly)
Accessories	One pair of furnace gloves
	One crucible steel tong
Optional	Exterior made of SS 304/SS316 (GMP model)
	30 Segment profile controller for heating and cooling rate
	PLC with Color HMI (data logging and USB Interface)
	RS485 with PC interface
	Data logger with PC/USB interface
	Calibration certificate
	Door safety switch
	Silicon carbide bottom plate
Caster wheels with brake	

Hot plate

Procedure for an experiment

Figure 2. Microwave solution combustion method with dye degradation.

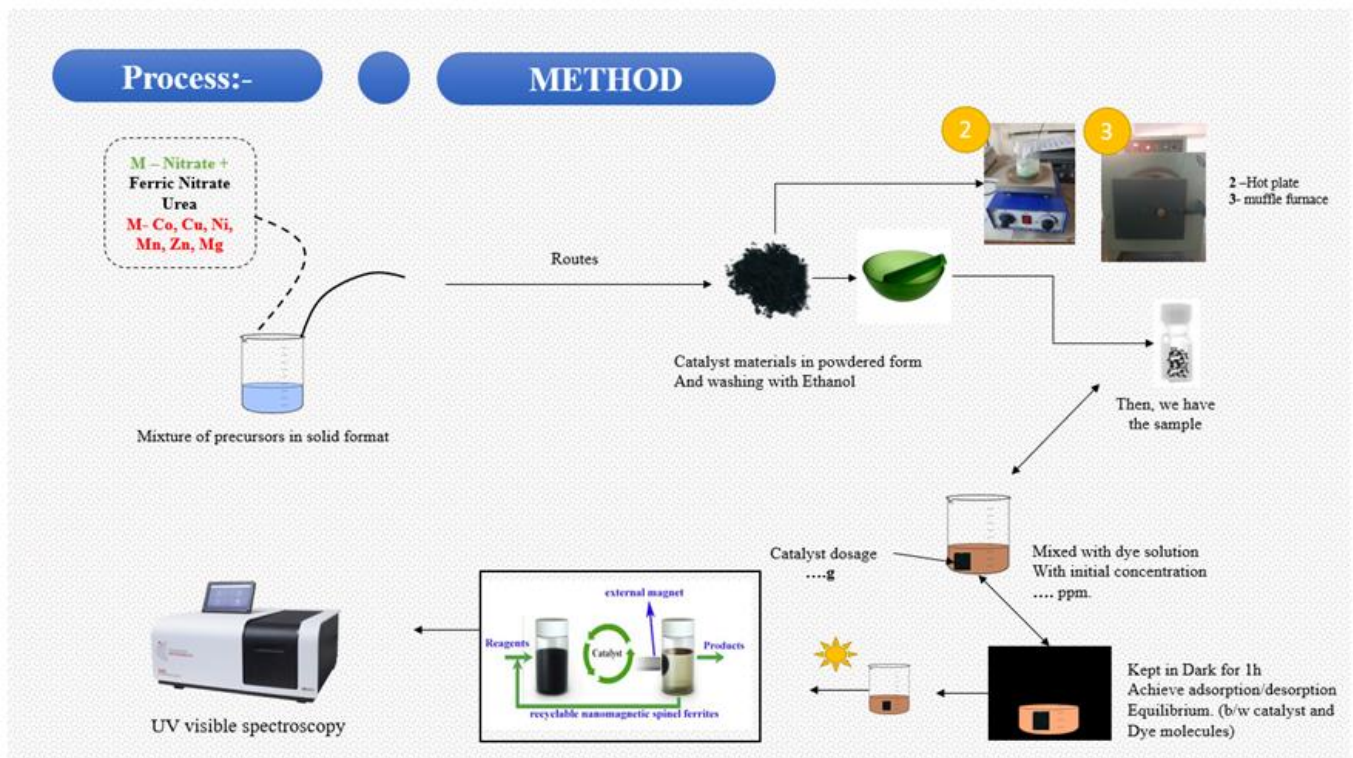


Required amounts of iron nitrate  $Fe(NO_3)_3 \cdot 9H_2O$ , mixture with divalent metal cation with fuel consisted reducing agent and it also capable for combustion reaction. The amount of mixture calculated by stoichiometry relation listed in Table 4 above. Then proceed to make propellant dissolved 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  $80\text{ }^\circ C$  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  $250\text{ }^\circ C$ , 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 (Figure 3).

Figure 3. Procedure of solution combustion synthesis experiment.



The various amounts of fuels with the precursors dissolved in distilled water. Normally can take 30 ml, 50 ml depend on the amount of material to be make. The combustion gaseous product was considered as CO<sub>2</sub>, H<sub>2</sub>O, and N<sub>2</sub>. But its depend on the combustion temperature. After the process proceed for reaction F/O ration equalized to 1 but sometimes 1.5 or >1 respectively. A brown dried gel combusted in semi closed system or can use types of muffle furnace for combustion. The temperature can be determined by TGA/DSC analysis (thermal deposition of powder). It can be 500°C, 600°C, 700°C- RRJET | Volume 11 | Issue 4 | April, 2022

1000°C. Then, dried powdered crushed by mortar with pestle.

## Photocatalysis

### 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  $\text{cm}^{-1}$  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 revels the presence of tensile strain in the composites. The X-ray density (dx) for each sample was calculated by the relation.

$$dx = \frac{ZM}{NV}$$

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 be investigated with VSM (Vibrational Sample Magnetometry) by sweeping 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 ratio  $M_r/M_s$  less than 0.5, according to Stoner–Wolffarth indicate the nanoparticles possess 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 calculated using Brown's relation.

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

It has been observed that anisotropy constant ( $k_1$ ) decreases at higher calculation temperature ( $^{\circ}C$ ) (Table 4).

**Table 4.** Literature survey on Magnetization properties.

Catalyst name	Synthesis methods	Fuel	Saturation magnetization ( $M_s$ ) – emu/g	Remanence magnetization ( $M_r$ ) – emu/g	Coercivity ( $H_c$ ) (Oe)
CoFe <sub>2</sub> O <sub>4</sub> /RGO	Solvothermal synthesis	-	41.98	25.4	6.41
ZnSeWO <sub>3</sub> eCoFe <sub>2</sub> O <sub>4</sub>	Wet impregnation	-	0.6503	-	1000.03
Co doped ZnAl <sub>2</sub> O <sub>4</sub>	Microwave combustion method.	Glycine	-	0.23	217.39
Zn <sub>1-x</sub> CoxAl <sub>2</sub> O <sub>4</sub>	Microwave assisted combustion method	Ethylenediaminetetraacetic acid	-	0	52.25
8% Co doped Fe <sub>3</sub> O <sub>4</sub> NPs	Co-precipitation	-	29.51	6.48	308.14
MgFe <sub>2</sub> O <sub>4</sub> (glycine)	Solution combustion method	Urea	27	-	51
NiFe <sub>2</sub> O <sub>4</sub>	Solution combustion synthesis	Glycine	59	13	95

CoFe <sub>2</sub> O <sub>4</sub> , Co <sub>0.5</sub> Mn <sub>0.5</sub> Fe <sub>2</sub> O <sub>4</sub>	Solution combustion synthesis	Oxalyl di hydrazide	76.1	-	-
Ni <sub>0.50</sub> Co <sub>0.50</sub> Fe <sub>2</sub> O <sub>4</sub>	Microwave combustion synthesis.	Glycine	33.3	14.3	941

### CONCLUSION

Microwave solution combustion or solution combustion method both works collimated each other. The great propellant discovery found in the microwave and easy reaction with less contact time to produce a suitable catalyst for dye degradation. Our study shows microwave comes in drawback with a large number of gases produced (NO<sub>x</sub>, CO<sub>x</sub>, NH<sub>3</sub> etc.) but it depends on the selected material for combustion and its produced by exothermic reaction with as in (CoFe<sub>2</sub>O<sub>4</sub>, NiFe<sub>2</sub>O<sub>4</sub>, MgFe<sub>2</sub>O<sub>4</sub>-spinel ferrites materials) and its produce good magnetization for easy separation with dye (either reactive dyes or others). Solution combustion assisted work the catalyst for reducing particle size with a certain amount to temperature (400-2000 °C) in muffle furnace during calcination and produce crucial separation. So, the microwave is a very effective work for dye degradation as an application.

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