

Green Sol-Gel Synthesis Method for $AxbyFe_2O_4$ ($X, Y = 0.1$ To 0.5 , Alternatively) Nanomaterial Changes in its Magnetic Properties and Utilized for Photocatalytic Applications

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Submitted: 25 Oct 2023

Accepted: 29 Oct 2023

Published: 04 Nov 2023

Citation: Dr K Suresh¹, and Rahul Jarariya^{2*} (2023). Green Sol-Gel Synthesis Method for $AxbyFe_2O_4$ ($X, Y = 0.1$ To 0.5 , Alternatively) Nanomaterial Changes in its Magnetic Properties and Utilized for Photocatalytic Applications, J of Physics & Chemistry 1(1). 01-10.

Abstract

Nanomaterials are evidence of usage in various fields of research in optics, electronics, bio medics, pharma industries, and wastewater treatment. Green Sol-gel synthesis is one of the processes that has gained popularity in recent years. It is based on the controlled odorless of alkoxides of silicon, Aluminum, and Transition metals $M(OR)_x$ like Titanium, Zirconium, Tungsten, Zinc etc. The presence or absence of substances with nonhydrolyzable bonds in the precursors, their concentration and ratio, the medium's pH, an acidic or basic catalyst, the presence of organic components, water-soluble polymers, and microorganism cells in the system, to name a few, all affect the structure of the forming sol-gel matrices. The plant-based NPs using "Ocimum tenuiflorum" based extract is an innovative way of a sustainable route for producing NPs without hazardous chemicals involved in this route. The hydrolysis and condensation reactions, which are typically regulated by the solution's pH, are the shape of the structure and condensation typically starts when hydrolysis is finished in the preceding perspective, however, when acid catalysis is used, hydrolysis advances more quickly than condensation. The NPs are suitably eligible to reduce the toxic effect of dye wastewater. The degree of dye attachment to the fibre material determines how much waste dye is produced. Anionic and Cationic dyes are two categories of synthetic dyes. In this study, we have covered metal nanoparticles that have been made from plant biomass during the past two decades as well as the numerous factors that determine the size and morphology of these particles. The use of NPs as a possible photocatalyst for the breakdown of organic dyes in solar radiation and the operating parameters for dye degradation

Keywords: Nanomaterials, Wastewater, Cationic Dyes, Ocimum Tenuiflorum, Sustainable, Photocatalyst, Biomass.

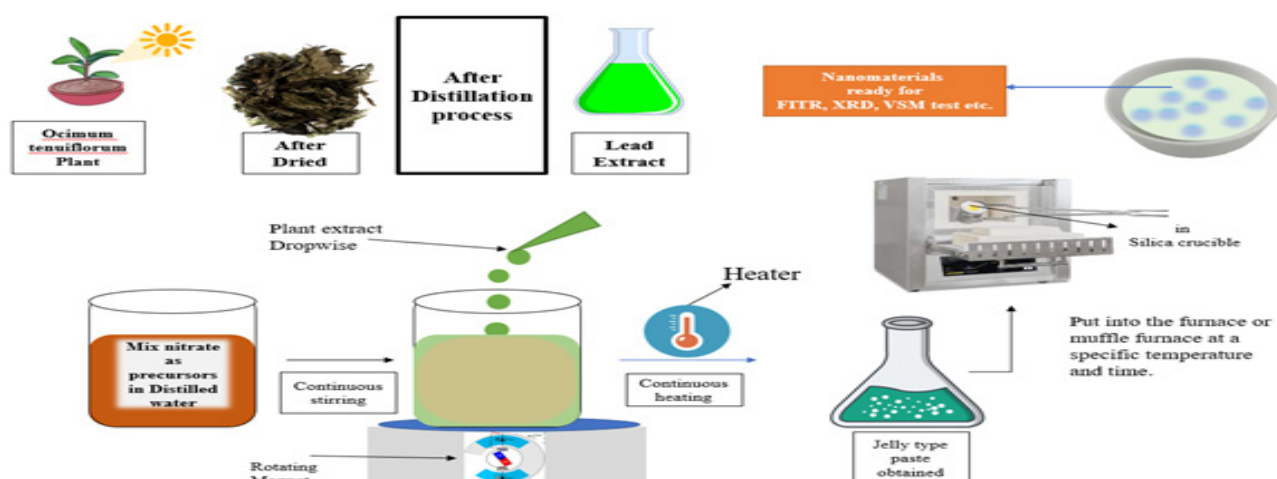


Figure 1: Nanomaterial Preparation Process

Introduction

Nano which means extremely small (10^{-9} m), to see the changed properties in nanoscale. Due to the physical and chemical changes, the materials are different in its structure, surface area, high thermal conductivity and extremely chemical reactive substances in it. In modern research, nanomaterials are widely used in various applications in the field of nanoscience and nanotechnology. Nanomaterials use in optical sensors, microwaves, magnetic drug delivery, storage devices. Spinel ferrite or doped spinel is an important structured material class of oxides with interesting properties and wide range of its applications. A spinel unit cell consists 56 ions, 32 O_2 , 24 are metals. Basic formula to known spinel is AB_2O_4 with FCC (Face Centered cubic structure) where A represent Divalent and B is Trivalent cations (ions or quadrivalent cations alike Aluminum, Cobalt, Iron Nickle, Zinc, Magnesium, Chromium etc. In this paper we are going to deal or manipulate to change its structure from Shifting with B – trivalent cations considered to change with its Iron (Fe) Doping to change in its magnetic hysteresis because when extracting molecules from alternative component so its easily separable. Doping spinel plays vital role in science technologies because of its magnetic and electrical properties with enhance physical and chemical changes. Moreover, the antiferromagnetic super exchange interaction between Fe^{2+} and Fe^{3+} ions control these features. The small-scale substitution of RE elements into the spinel ferrite lattice results in RE^{3+} - Fe^{3+} interactions. Doping of Fe^{+3} ion into structure of spinel ferrites induces strain and may cause distortion in structure, therefore modifies the structural, electrical, morphological, and dielectric properties. Literature suggested that the incorporation of rare earth ions in most of the spinel ferrites would result in improvement of structural properties. In discuss about its application, nanomaterials wide range of application are drug delivery, cancer treatment, anti-bacterial, cosmetics, catalysis etc. Lot of research suggested nanomaterials implement on wastewater treatment of catalytic activity.

Catalysis will provide the sites for the reactants to be activated leading to the formation of the products. Nano-catalysts have a nano-scale dimensions, which could be used to be overcoming mass transfer limitation and reducing the cost of biofuel production. Nanoscale catalyst

can be divided into various types based on carbon, graphite, and metal oxides. Nano-catalyst enhance the selectivity of the reactions at a lower temperature with higher recycling rates and less energy consumption. Hence, there are more effective alternatives when compared to conventional catalyst for the efficient production of biofuels production with higher yields. Three to five times higher conversion ratios enhance of surface area of that catalyst.

Industrial Concerns

Dyes are one for the most hazardous pollutants used in may industrially sector, such as the textiles, clinical substances, and food cosmetics industries. They are difficult to degrade by conventional methods. According to WHO (World Health Organization) maximum usage to chemicals exceed due to this reason. Our paper telling benefits to use Nanomaterials in wastewater treatments and provide better sustainable way to achieve desired nanoparticles in it. These plant-based nanomaterials widely acceptable for wastewater treatment applications and utilized in several sectors.

Production Method for Spinel Ferrite Nanomaterials

Many methods, including as hydrothermal, microwave solution combustion, solution combustion, sol-gel auto-combustion, co-precipitation, micro-emulsion, etc., can be used to create spinel ferrites nanomaterials. The earlier techniques, however, take a lot of time and can potentially harm herbivores and the environment. The Green Sol-gel method can be used to create spinel to illustrate this idea. Even if it takes time, it is suitable for handling.

Green Sol – Gel Method

Sol Gel Combustion Synthesis: This method is well known for less time consuming, high sintering temperatures, and fast heating rates. There are numerous advantages to accept that low cost, low temperature, easy handling, desired product, in previously, Nickle-copper nanomaterials investigated by using XRD (X-ray diffraction), Scanning electron microscopy, Fourier Transform Infrared Spectroscopy, Scanning Electron Microscopy (SEM). Aluminum doping in place of iron ions can reduce dielectric loss with increase in the electrical resistivity and decrease of saturation magnetization were investigated. In previously, the combination of two to more divalent metal ions are referred as the mixed ferrite used for implementing the ferrites. The distribution of the

cations between tetrahedral and octahedral sites affects the surface characteristics of the mixed ferrites. Additionally, it is anticipated that additional compositions of two distinct metal ferrites with hard and soft magnetic natures will fine-tune the features. The prepared sample investigated found high saturation magnetization, where the MNPs increases with $x=0.5$. For example, the particles of 161 and 175 AM²/kg for Zinc ferrite and Magnesium doped ferrite observed respectively [1, 2, 3]. For inverse spinel they investigated about the morphology and structure created by SGAC method.

The Advantages of Sgac Method

Cost less, high purity, high crystallinity NPs. By keeping magnetic resonance, the method is utilized for great performance.

Sources of Green Nanomaterials

As science and engineering advance into a new era, the application of green nanomaterials and green nanotechnology is of utmost importance. The vastness, intelligence, and futuristic nature of human society are geared for a new generation. Plant extracts, biopolymers, vitamins, proteins and peptides, sugars, and plant extracts are just a few of the chemical compounds found in nature that act as reducing agents. The most thoroughly researched so far are plant derivatives. The production of metal nanoparticles, valuable in electronics and medicinal applications, utilizing plant extracts as reducing agents, is one area of special scientific accomplishment. Another class of natural resources utilized for the manufacture of metal nanoparticles is biopolymers. These polymers' carbohydrate molecules have previously been put to use in a variety of applications and can be utilized to produce nanoparticles on a massive scale.

Microwave Solution Combustion Method

It is fast and auto combustion technique by using microwave. In previously, Raga scientific microwave we used. Magnesium ferrite prepared by microwave solution combustion method. This method implements in less process reaction time, and easy handling equipment. However, due the irradiation loads the materials destroys the shape and structure and may the harmful effects. By this irradiation, chamber closeness is important because its continuous reaction. If may not, then it damages human brain cells. The microwave frequency should be 2.45 GHz, and temperature should be maintained by temperature sensor (inside). Due to these safety measurement process will implement. Otherwise, suffer from toxicity. The major concerns in microwave system i.e., gases (CO₂), NH₃ release from top of the microwave. Harmful effect inhalation during self-ignition, the materials change its color from pink to brown due to microwave inside temperature.

What is Green – What is Sol Gel Synthesis?

According to the previous research reports, green word is used for leaf extract from Brassicaceae family, whereas the sol gel was adopted in the case of chemical synthesis. The particles were developed by sol gel method and analyzed via UV-vis, EDX, XRD, SEM techniques etc. This synthesized NPs was assessed against bacterial and fungal strains by disc diffusion method. Acute sized NPs shown promising results for antimicrobial activity and zones of inhibition were acute range (10-20 nm) [3] whereas streptomycin (standard drug) showed the zone of inhibition of 25 mm. The green sol gel synthesis become popular to use for the fabrication of ZnO NPs as an antimicrobial agent.

Spinel ferrites	Method	Materials	Characterization	Ratio	pH	Investigated	References
NiFe ₂ O ₄	Sol-gel and Hydrothermal	Ni (NO ₃) ₂ ·6H ₂ O, Fe (NO ₃) ₃ ·9H ₂ O, NiCl ₂ ·6H ₂ O, FeCl ₂ ·6H ₂ O, NaOH, (CH ₂ OH) ₂	XRD, FTIR, VSM, SEM	Hydrothermal (1:2:8), Sol gel (2:1)	Hydrothermal (pH=8.0), Sol gel (At material pH)	Structural, dielectric, magnetic (M _s /M _r , /H _c) properties	[4]
Ni _{0.5} Cu _{0.5} Al _x Fe _{2-x} O ₄ , (x=0.0, 0.2, 0.4, 0.6)	Sol-gel auto-combustion route	Cu (NO ₃) ₂ ·3H ₂ O, Fe (NO ₃) ₃ ·9H ₂ O, Ni (NO ₃) ₂ ·6H ₂ O, Al (NO ₃) ₃ ·9H ₂ O, Citric acid monohydrate	XRD, FTIR, SEM	-	-		[5]
NiCoFe _{2-x} Al _x O ₄ (x=0 to 0.4)	Sol-gel auto-combustion	cobalt (II) nitrate (Co (NO ₃) ₂), nickel (II) nitrate (Ni (NO ₃) ₂), iron (III) nitrate hexahydrate (Fe (NO ₃) ₃ ·9H ₂ O), and aluminum (V) nitrate Al (NO ₃) ₃ ·9H ₂ O, FEI NOVA 450 Nano SEM	XRD, FTIR, SEM	1:2	-	High conductivity and potential dielectric properties	[6]
Ni _{0.4} Cu _{0.3} Zn _{0.3} Ag _{0.4x} Mn _{0.3x} Cr _{0.3x} Fe _{2-x} O ₄ (x = 1/4 0.0, 0.05, 0.10, 0.15)	Sol-gel auto-combustion technique	Ni (NO ₃) ₂ ·6H ₂ O, Cu (NO ₃) ₂ ·6H ₂ O, Zn (NO ₃) ₂ ·6H ₂ O, Cr (NO ₃) ₂ ·6H ₂ O, Mn (NO ₃) ₂ ·6H ₂ O, Ag (NO ₃) ₂ ·6H ₂ O, Fe (NO ₃) ₃ ·9H ₂ O,	XRD, FESEM, VSM		7	Magnetic and electrical traits	

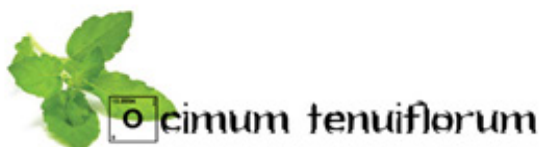
		Citric acid and ethylene glycol					
Ni _{0.4} Zn _{0.45} Co _{0.15} Fe ₂ O ₄ /FeSiAl	Sol-gel auto combustion method	Fe (NO ₃) ₂ ·9H ₂ O, Zn (NO ₃) ₂ ·6H ₂ O, Ni (NO ₃) ₂ ·6H ₂ O, Co (NO ₃) ₂ ·6H ₂ O, C ₆ H ₈ O ₇ ·H ₂ O, 0.1g/ml of phosphoric acid or ethanol.	VSM, DTA-TG, XRD				[7]
Cu _{1-x} Ni _x Fe ₂ O ₄ (0,0.5,1)	Sol gel method	0.5 M of Ni (NO ₃) ₂ ·6H ₂ O, 0.5 M of Cu (NO ₃) ₂ ·3H ₂ O, and 1 M OF Fe (NO ₃) ₃ ·9H ₂ O	XRD, FTIR, SEM, UV-vis, VSM				[8]
NiCoFe _{2-x} Al _x O ₄	sol-gel auto-combustion	cobalt (II) nitrate (Co (NO ₃) ₂), nickel (II) nitrate (Ni (NO ₃) ₂), iron (III) nitrate hexahydrate (Fe (NO ₃) ₃ ·9H ₂ O), and aluminum (V) nitrate Al (NO ₃) ₃ ·9H ₂ O	XRD, FTIR, SEM	1:2			[9]
ZnO NPs	Green versus sol-gel synthesis	0.1 M Zn (NO ₃) ₂ · 6 H ₂ O (100 ml), NH ₄ OH	XRD, SEM, VSM, FTIR	-	5.0	Antifungal and antibacterial potential	[3]
Mg _{0.5} Zn _{0.5} FeMnO ₄	Green sol-gel synthesis	Mn (NO ₃) ₂ ·4H ₂ O, Mg (NO ₃) ₂ ·6H ₂ O, Fe (NO ₃) ₃ ·9H ₂ O, Zn (NO ₃) ₂ ·6H ₂ O	XRD, SEM, FTIR	1:2	6.0	Decolorized RB21 dye	[10]


Table 1: A Review Based on Green Synthesis Nanomaterials

Review on Ocimum Tenuiflorum

“Ocimum Tenuiflorum” is also known as holy basil “Tulsa”. One of the holy plants followed part of mint family. In India, many accreditations of “Tulsa” which is used on ceremonies, hymns, worships etc. majorly used in foods items like poha, vegetables, others. Ocimum tenuiflorum grown in many homes outside in front of the main gate, it covered in four side structured earthen flowerpot. According to Hindu culture, Ocimum tenuiflorum is giving foster meditation and purifies blood and protect from certain concerns. Holy basil is used in various major part like Devoting, Hymns mala, weeding ceremonies, Vishnu pooja.

There are large benefits about Ocimum tenuiflorum to use in foods preparation although it is used in tea to reduce stress, weight loss, reduce cancers lung concerns, clean throat etc. In modern advantages Ocimum tenuiflorum use in vegetables salads to improve taste. While not being the Hyssopus officinalis, which is native to Palestine, Ezov, the hyssop of the Bible, may have been a caper or savoury that was once used in the ritual bathing of lepers. [11]. The origin of religious Ocimum tenuiflorum species conducted using chloroplast genome sequences, previous study submitted of this originality from Central University of Punjab, Bathinda, they mentioned that North central India has basic rights. Oleanolic acid, rosmarinic acid, eugenol, carvacrol, linalool, beta-caryophyllene, ursolic acid trace compounds present in Ocimum tenuiflorum. A perennial aerodynamic plant of the Lamiaceae genus.



Chemical Composition	Uses	Common Names	Properties		Present in Ayurvedic Medicines
Carvacrol	Fever	Krishna Tulasi (Karnada)	Rasa (Taste)	<i>Bitter, Astringent</i>	Vilwadi Gulika
Caryophyllene	Common cold	Tulsi (Hindi)	Guna (Qualities)	<i>Light, Dry, Sharp</i>	Manasamitra Vatakam
eugenol	sore throat	Tolasi (Tamil)	Veerya (Potency)	<i>Hot</i>	Maha Jwarankush ras
linalool	Headache	Tulasi (Telugu)	Vepaka (Post digestion effect)	<i>Pungent</i>	Muktadi Maharjan
ursolic acid	Eye problems	Tulasi (Sanskrit)	Karma (Pharmalogical activity)	<i>Kapha, Vata</i>	Mukta Panchamirit ras
Part for use	Dental problems	Holy Basil (English)	Prabhava (Nutritional compounds)	<i>Vitamin A & C, Calcium, Iron, Magnesium, Phosphorous, Potassium, Sodium, Zinc, Eugenol, Manganese.</i>	
Flowers	Skin disorder	Leaf		Flower	
Leaves	Insect bites	Kind	<i>Simple</i>	Type	<i>Bisexual</i>
	Kidney Stone	Shape	<i>Opposite</i>	Size	<i>Receme</i>
Mode of Propagation: Seed and Cuttings	Common growing areas: Tropical area, Subtropical area, warm filed.	Feature	<i>Leaves 1.3- 3 x 0.6-2 cm, elliptic-oblanceolate, obtuse at both ends, margin coarsely serrate, puberulous; petiole to 2 cm long, covered with reddish-purple hairs</i>	Color and Compostion	<i>Pink/White</i>
Good time				Stamen	<i>4</i>
<i>September to February</i>				Other information	<i>Racemes to 11 cm long, generally simple, occasionally branched at base; pedicel c. 2 mm long. Flowering throughout the year</i>

https://en.wikipedia.org/wiki/Ocimum_tenuiflorum

@Stubborn engineer

Figure 1: All Information About Ocimum Tenuiflorum (Herb)

Sr. No.	Materials Extracts can be used to prepare NPs
1	Raphanus sativus Linn. Var. longipinnatus Bailey (Red radish)
2	Raphanus sativus linn. (White Radish)
3	Brassica rapa linn. (Turnip)
4	Brassica campestris linn. Var. sarson parin (Saag)
5	Brassica oleracea var. botrytis linn. (Cauliflower)
6	Holy basil (Tulsi)
7	Aloe vera
8	Camellia sinensis
9	Curcuma longa
10	Brassica juncea
11	Cinnamomum camphora
12	Citrus limon
13	Ludwigia adscendens
14	Pelargonium roseum
15	Rhododendron dauricum
16	Terminalia catappa
17	Pinus thunbergii extracts

Table 1: Preparation of NPs by extracts

Pros and Cons of Green Synthesis

In green synthesis of NPs, the precursors are the most expensive followed by the substrates which must be procured and functionalization step of the nanoparticle is eliminated when we use a bio-nano catalyst unlike in physical synthesis. Phytochemicals, and enzymes act as stabilizers in plant and microbial based synthesized nanoparticles. Many researchers have explained that green synthesized nanoparticles play an important role in the medical field for diagnostic applications. It concluded that green method of nanoparticles synthesis shown better antibacterial, antifungal, anti-parasitic activities like copper NPs fabricated using malva synthesis have antibacterial, antifungal, and antiparasitic activities.

Nanoparticles can be exploited for cleaning hazardous waste sites and pollutants. Similarly, other workers have reported that iron NPs can be used for treating water and soil remediation. Bacteria and diatoms produced magnetic and siliceous materials which were reported to be useful for optical coatings. Bio-synthesized TiO₂ by Psidium guajava showed significant antibacterial and antioxidant activity (like onion peel) of CuO NPs and ZnO-NPs against E. coli, B. subtilis, and S. aureus was higher when compared to chemically synthesized nanoparticles. The size and shape of the nanoparticles and the compound involved in their synthesis are unknown.

Future Vision and Sustainability

The future of human science depends on both imagination and inventiveness. Academic rigour today is characterised by environmental management and sustainable development. Environmental engineering and management are advancing quickly, are crucial for a growing number of human activity sectors, and are essential to the accomplishment of sustainable development. The availability of fundamental human needs like energy and water is crucial to the development of human civilization and scientific rigour. Today, there is a great deal of prudence, scientific cleverness, and scientific fortitude

being used to the purification of water and the treatment of wastewater. Remediation of heavy water groundwater is a real challenge because there are now so few answers to the enormous problem of arsenic poisoning of groundwater. With the passage of time and history, the difficulties of scientific endeavor in the field of water science and technology need to be envisioned and reconstructed. A new visionary era in green nanotechnology and green chemistry is being ushered in by the scientific struggles, brilliance, and massive requirements of the science of sustainability.

Photocatalytic Activity

Through MG and MB degradation under UV light, the photocatalytic activities of the pure and co-catalytic silver-modified films were studied. Previous studies investigated the parallel sol gel technique processes for the synthesis of the TiO₂ and ZnO catalysis in order to analyze the effect of the co-catalytic modification. A linear regression was used to assess the apparent first-order reaction rate constant using the experimental data. Each correlation coefficient's (R²) value exceeded 0.947. It is widely known that a variety of parameters, including the synthesis process, light, illuminations, crystalline structure, surface area, etc., affect the photocatalytic efficiency of co-catalytically modified semiconductors. Without the pure and co-catalytic silver modified semiconductor sheets, there was no bleaching of the malachite green solution.

The capacity of silver to trap electrons may help to explain the beneficial effect of co-catalytic modification with silver ions on the effectiveness of TiO₂ and ZnO for the photodegradation of the dyes. The rate constant of photocatalysis is represented by the slope of the linear fits to the logarithmic scale.

Eventually, utilizing the sol-gel method and co-catalytic modification of Ag, it was successful to tune the electronics structure of the TiO₂ and ZnO samples. Furthermore, their extraordinary efficiency motivates us to use this test in the presence of ultraviolet light on another organic contaminant. The decolorization of MB dye or other dyes was explored for the photocatalytic activity of the sol gel films.

Applications

Bioenergy

Developing clean and efficient energy technologies has become the need of the hour due to the ever-increasing energy demand. Nanotechnology application in biofuel production mainly focuses on breaking down the feedstock, and improving more efficiently, decreasing the transportation cost of feedstock. In biofuel production, economic, covalent linking of an enzyme to a support is the more interesting method of immobilization which of an enzyme to a support is the most interesting method of immobilization which is based on activation of chemical groups to support its reaction with protein nucleophiles. With the dwindling energy sources, the problem of energy crisis looming at large, all countries are looking for alternative's sources of their energy needs. Indians have primarily dependent on conventional energy sources, such as fossil fuels such as coal and petroleum, which has caused rapid depletion of fossil fuels with significant effects on the environment [12]. Potential bioenergy research can benefit from the use of nanomaterials. Because they are self-existent, omnipresent, and leave no harmful byproducts or residues, bioresources are renewable

Nanoparticles used for biofuel production	Biofuels
SiO ₂ and TiO ₂	Biogas
KF/CaO nanocatalyst	Biodiesel
Gold nanoparticles	Ethanol
Magnetic nanoparticles	Sugars
Cobalt nanoparticles	Biodiesel
Nano-iron oxide (Fe ₃ O ₄ NPs)	Methane
Nano catalysts and sulfonic acid cobalt spinel ferrite magnetic nanoparticles	Biogas

Table 2: Nps for Biofuel Synthesis

An inexpensive substance that is effective in treating wastewater from the textile industry is rice husk-based biomaterial (RHBB), a waste product from the rice milling industry. According to studies, the textile industry can use up to 200 L of water for every kg of color. As a result, a lot of water is produced, much of it heavily contaminated by dyes, heavy metals, complex salts, acids, etc. This will, as anticipated, cause pollution-related issues for the receiving water bodies. According to several studies reviewed, this problem has been addressed using a variety of techniques, including membrane separation, advanced oxidation, chemical oxidation, ozonation, adsorption, coagulation, and catalysis, some of which have inherent difficulties, are ineffective, or are environmentally unfriendly. Now, with great present of green sol gel generate NPs works truly well in presence of *Ocimum tenuiflorum* leaves extract molecules in spinel ferrite structure.

Conclusion

The gel that is deposited reduces stiffness even though green sol is applied to (*Tulsi*) leaves to diminish the magnetic moment. The media is disseminated at the bottom of the mixture; Ms, Mr. are strong enough to separate from the magnetic moments of nanomaterials, and Neodymium magnets are used to achieve better magnetic separation. Green Sol Gel's photocatalytic activity lasts for a shorter period. Green sol route decolorized dye as fast process. This strategy economically viable, wide range, non-toxic, easy to scale up, volatile organic solvents, surfactants. Our conclusion is that green sol production of catalyst with *Ocimum tenuiflorum* is superior to other methods. Some data for the application of dye degradation are still pending from SEM, TEM, VSM, XRD, and UV spectrophotometer. In the past, CR dye was utilized to its greatest capacity, and with the application of natural gel, they were able to remove 97% of the dye's color. Now, our work demonstrates that dye's reversibility to degrade increases with an increase in its antibacterial and antifungal activities, removing more than 99% (approx.) of the dye in effluent.

Acknowledgments

This work was supported by Professors at M.A.N.I.T Bhopal, MP, India. Chemicals procured from L.N.C.T., Bhopal for Nanomaterials generation [13-19].

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