



INTERNATIONAL JOURNAL OF CREATIVE RESEARCH THOUGHTS (IJCRT)

An International Open Access, Peer-reviewed, Refereed Journal

Synthesis Of Fe_3O_4 Nanoparticle By Solution Combustion Using Taguchi Optimization Method

Nandkishor pandey and Sandesh Jaybhaye

Nanotech Research Lab, Department of Chemistry, B. K. Birla College, Kalyan, India

Abstract:

Ferrite Nanoparticles is magnetic in nature due to these they are used in many fields like biomedical field (in magnetic resonance imaging and magnetic hyperthermia etc.), Due to its magnetic properties it used in drug delivery system and as a heterogeneous catalyst in organic synthesis. It is prepared from different methods like Green Method, Chemical Precipitation Method and Solution Combustion method etc. In this work, Solution combustion Synthesis of ferrite (Fe_3O_4) nanoparticle is carried out. A Taguchi optimization method is used for the synthesis of ferrite nanoparticle. By considering all four variables we find that each variables affect the chemical composition and magnetic properties of synthesized ferrite nanoparticle. In this work, we consider Taguchi optimization for synthesis which give better quality ferrite nanoparticles. Ferrite Nanoparticles has antibacterial activity.

Keywords: Ferrite Nanoparticles (FNPs), Taguchi optimization, Solution Combustion Synthesis

Introduction:

Ferrite Nanoparticles has magnetic properties hence it is present in the class of magnetic nanoparticles(MNPs) due to its interesting properties it is used in various fields like biomedical to industrial. Magnetic material is used in many technological applications. The variables which is used during the synthesis may affect the geometry of synthesized material like size which is reduced to nanoscale has many advantages then their bulk form. The all these properties show that these nanomaterials is used in medical diagnosis and treatment in medical field and it also used as sensors in energy sector.

Ferrite Nanoparticles is found in three major forms such as, Magnetite (Fe_3O_4), Maghemite ($\gamma\text{-Fe}_2\text{O}_3$), and Hematite ($\alpha\text{-Fe}_2\text{O}_3$). The maghemite phase of ferrite is not practically useful for different applications. It is unstable compare to hematite form of ferrite but it can exist along with other forms. The hematite is the most stable form of ferrite nanoparticle and highly used forms. The solution combustion synthesis of ferrite Nanoparticles shows following reaction,



The Taguchi method was developed by Dr. Genichi Taguchi of Japan. He maintained that variation during the experiment. Taguchi developed a method for designing experiments to investigate how different parameters affect the mean and variance of a process performance characteristic that defines how well the process is functioning.

These ferrite nanomaterials is prepared by a different methods, such as co-precipitation thermal decomposition, sonochemical or solvothermal. Most of these methods give good results in terms of low particle size and morphology.

At present time, the sol-gel and solution combustion technique is good option due to its simplicity and control over the product characteristics and parameters used during synthesis. In these work, four variables is consider at a time and observe their interaction . The effect of each variables on the chemical composition and magnetic properties is study by different method. The FNPs are characterization by UV spectra, X- ray diffraction and FTIR spectra.

Materials and Methods:

Materials: The metal salt precursors used for the synthesis of Fe_3O_4 Nanoparticle were iron nitrate [$\text{Fe}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}$, Research lab], with citric acid, urea and glycine (Research lab) as a fuels. Distilled water was used as the solvent.

Experiments design:

The experiment design which is a used for synthesis of Fe_3O_4 Nanoparticle by solution combustion method using Taguchi optimization method and check which parameters affect the synthesis of Fe_3O_4 Nanoparticle. In this work, a Taguchi optimization method is used in which four parameters were used such as fuels, temperature, time and ratio as shown in the table 1.

	Levels		
Optimization parameters	1	2	3
FUEL	Citric acid	Urea	Glycine
TEMP	400	500	600
TIME	30	60	90
RATIO	1:1	1:2	2:1

Table 1

Synthesis:

For the synthesis of (Fe₃O₄) nanoparticles by the solution combustion method, Weight amount of ferric nitrate was first dissolved in 5 mL of distilled water with constant stirring at room temperature. Fuels is also dissolve in 5 ml distilled water. After this, Mix the both the solution carefully till homogeneous solution was obtained. Heat these homogeneous solution at 400 °C for 30 in a furnace to obtained the FNPs. The obtained product was further grind to obtained Fe₃O₄ Nano powder.

Characterization:

The synthesized FNPs are characterized by different instruments. XRD measurement of FNPs give information about whether synthesized nanoparticle is crystalline or amorphous measurements. XRD measurement also help to determine chemical composition, crystal structure and particle size of synthesized nanoparticles. The Bragg's Law ($n\lambda = 2d \sin \theta$) equation is used for size determination of ferrite nanoparticles. FNPs are also analyzed by UV and FTIR spectrum which gives information about the absorbance maxima and function group present in synthesized ferrite nanoparticle.

Result and discussion:

The results obtained from characterization of the FNPs show following results. The temperature range have been set between 400 and 600 °C, whereas the time varies from 30 min to 90 minutes. This first samples (1:1) been characterized by X-ray diffraction (XRD) to determine their crystal structure, chemical composition and particle size. The characteristic Fe₃O₄ peak at around 32.2046° is present in all of the samples, but it seems clear that the peak at 24.1677°, corresponds to Fe₃O₄ nanoparticles.

Level	Fuel	Temperature in °C	Time in Minutes	Ratio	Average particle size in nm
1	1	400	30	1:1	33.32
2	1	500	60	1:2	34.82
3	1	600	90	2:1	37.67
4	2	500	30	2:1	34.87
5	2	600	60	1:1	43.20
6	2	400	90	1:2	43.29
7	3	600	30	1:2	36.33
8	3	400	60	2:1	34.92
9	3	500	90	1:1	37.73

Table 2

Debye – Scherrer equation for the calculation of particle size is

$$D = (k \lambda / \beta \cos \theta)$$

Where,

K is the Scherrer constant,

D is the size of the particle,

K is known as the Scherer's constant (K=0.94),

λ is wave length of the X-ray beam used (1.54,184 Å),

β is the Full width at half maximum (FWHM) of the peak and

θ is the Bragg angle

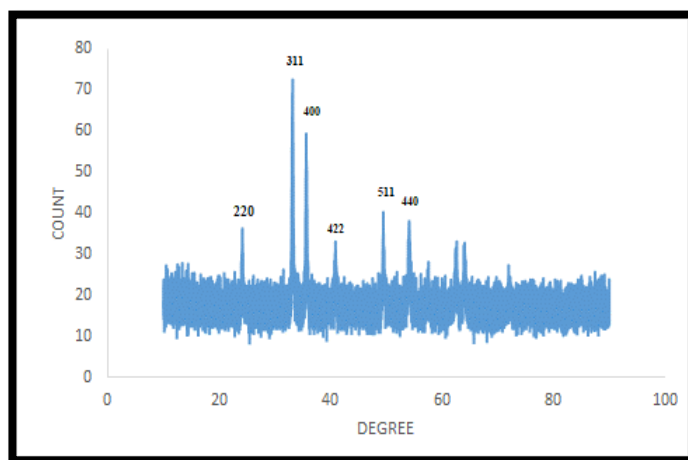


Fig.1 X-ray diffraction Fe₃O₄ Nanoparticles

Taguchi smaller the better calculation:

Ferrite nanoparticle synthesis by solution combustion using the Taguchi optimization method considering four parameters such as fuel, temperature, time, and ratio after carrying out 9 syntheses by considering all parameters, we obtained data and these data are used to calculate deviation and FOE% we obtained that fuel parameter affects more than the other parameters.

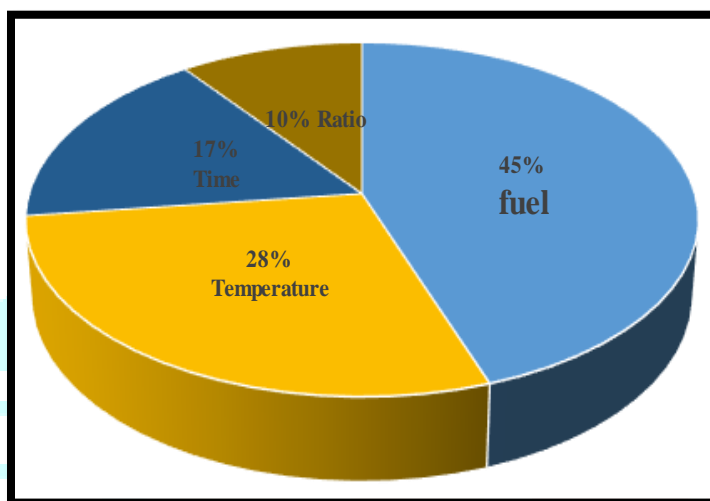


Fig.2 FOE% of Ferrite nanoparticles synthesis by Taguchi method

The above graph shows that four parameters affect the synthesis, but fuel parameters affect more (45%) than the other parameters like Temperature (25%), Time (17%), and, Ratio (10%). But we used three types of fuels hence we plot a deviation bar graph to know which fuel, temperature, time, and ratio affect more on ferrite nanoparticle synthesis.

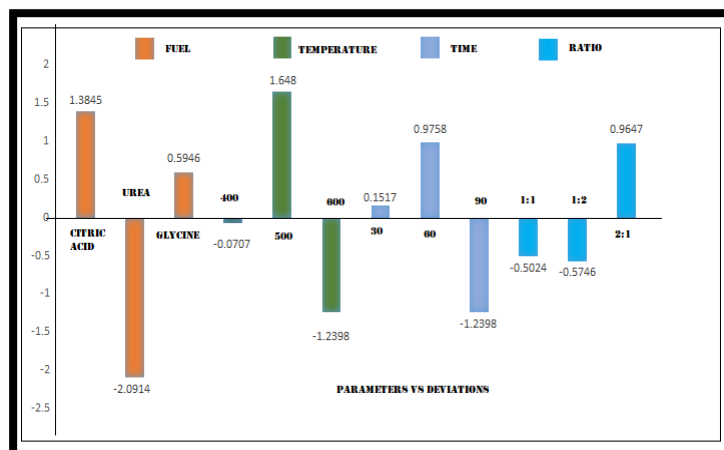


Fig.3 Deviation vs Parameters of FNPs by Taguchi method

The above graphs show that the fuel citric acid affects the synthesis more than the other fuel. The temperature at 500⁰C is best for FNPs, the time at 60 minutes and the ratio of 2:1 is best for FNPs synthesis. After considering all of the described parameters, we synthesized FNPs again and carried out its XRD for particle size determination.

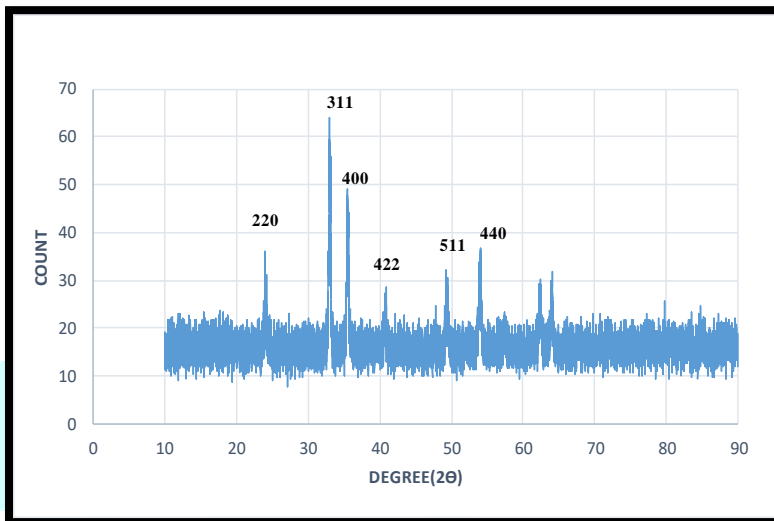


Fig.4 X-ray diffraction Fe₃O₄ Nanoparticles

Fourier Transform infrared spectra:

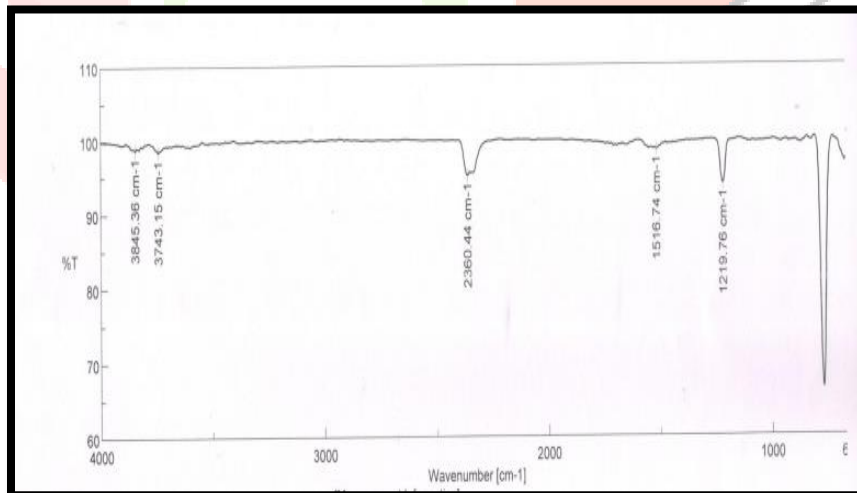


Fig.5 FTIR spectra of synthesized ferrite nanoparticle

The FTIR spectra show that the peak at 3443.15 cm^{-1} is due to O–H group (stretching vibration) which is come from water molecule which is present in Ferrite nanoparticles. The absorption peaks at 2360.44 , 1516.74 , 1219.76 , and 1020.54 cm^{-1} is due to distilled water which is used as a solvent. The absorption peaks at 695.29 and 468.80 cm^{-1} is due to the Fe–O bond vibration in Fe_3O_4 nanoparticles.

CONCLUSION:

In this paper, the ferrite nanoparticles have been synthesized by means of a simple solution combustion method using Taguchi optimization method. The process of synthesis is carried out using different parameters at different conditions. A Taguchi optimization analysis, by using different parameters like fuel, ratio, time, and temperature and its affects on the nanoparticles properties. Moreover, we observe that the Fuel parameter shows more impact on the final properties of FNPs than the other parameters. The synthesized FNPs using Citric acid as fuel at $500\text{ }^\circ\text{C}$ for 60 minutes shows that the size of the synthesized ferrite nanoparticles is 34.63 nm .

References:

- (1) Niemirowicz, K.; Markiewicz, K. H.; Wilczewska, A. Z.; Car, H. Magnetic Nanoparticles as New Diagnostic Tools in Medicine. *Adv. Med. Sci.* 2012, 57, 196–207.
- (2) Gao, Y.; Lim, J.; Teoh, S.-H.; Xu, C. Emerging Translational Research on Magnetic Nanoparticles for Regenerative Medicine. *Chem. Soc. Rev.* 2015, 44, 6306–6329.
- (3) Jozefczak, A.; Kaczmarek, K.; Hornowski, T.; Kubovc' íkova', M.; Rozynek, Z.; Timko, M.; Skumiel, A. Magnetic Nanoparticles for Enhancing the Effectiveness of Ultrasonic Hyperthermia. *Appl. Phys. Lett.* 2016, 108, No. 263701.
- (4) Shi, X.; Xiang, C.; Liu, Y.; Lin, H.; Xu, Y.; Ji, J. Preparation and Evaluation of Chitosan/ β -Cyclodextrin Magnetic Nanoparticles as a Photodegradable and Hydrophobic Drug Delivery Carrier. *J. Appl. Polym. Sci.* 2017, 134, No. 45076.
- (5) Kamzin, A. S.; Ranjith Kumar, E.; Ramadevi, P.; Selvakumar, C. The Properties of Mn–CuFe₂O₄ Spinel Ferrite Nanoparticles under Various Synthesis Conditions. *Phys. Solid State* 2017, 59, 1841–1851.
- (6) Chandra, S.; Patel, M. D.; Lang, H.; Bahadur, D. DendrimerFunctionalized Magnetic Nanoparticles: A New Electrode Material for Electrochemical Energy Storage Devices. *J. Power Sources* 2015, 280, 217–226.
- (7) Kovalenko, A.; Singh Yadav, R.; Pospisil, J.; Zmeskal, O.; Karashanova, D.; Heinrichova, P.; Vala, M.; Havlica, J.; Weiter, M. Towards Improved Efficiency of Bulk-Heterojunction Solar Cells Using Various Spinel Ferrite Magnetic Nanoparticles. *Org. Electron.* 2016, 39, 118–126.

- (8) Galloway, J. M.; Talbot, J. E.; Critchley, K.; Miles, J. J.; Bramble, J. P. Developing Biotemplated Data Storage: Room Temperature Biomineralization of L10 CoPt Magnetic Nanoparticles. *Adv. Funct. Mater.* 2015, 25, 4590–4600.
- (9) Ohkoshi, S.i.; Namai, A.; Yoshikiyo, M.; Imoto, K.; Tamazaki, K.; Matsuno, K.; Inoue, O.; Ide, T.; Masada, K.; Goto, M.; et al. Multimetal-Substituted Epsilon-Iron Oxide ϵ -Ga_{0.31}TiO_{0.05}-CoO_{0.05}Fe_{1.59}O₃ for Next-Generation Magnetic Recording Tape in the Big-Data Era. *Angew. Chem., Int. Ed.* 2016, 55, 11403–11406.
- (10) Salavati-Niasari, M.; Mahmoudi, T.; Sabet, M.; HosseinpourMashkani, S. M.; Soofivand, F.; Tavakoli, F. Synthesis and Characterization of Copper Ferrite Nanocrystals via Coprecipitation. *J. Cluster Sci.* 2012, 23, 1003–1010.
- (11) Kanagaraj, M.; Sathishkumar, P.; Selvan, G. K.; Kokila, P.; Arumugam, S. Structural and Magnetic Properties of CuFe₂O₄ As Prepared and Thermally Treated Spinel Nanoferrites. *Indian J. Pure Appl. Phys.* 2014, 52, 124–130.
- (12) Moshtaghi, S.; Ghanbari, D.; Salavati-niasari, M. Characterization of CaSn(OH)₆ and CaSnO₃ Nanostructures Synthesized By a New Precursor. *J. Nanostruct.* 2015, 5, 169–174.
- (13) Sharifi, I.; Zamanian, A.; Behnamghader, A. Synthesis and Characterization of Fe_{0.6}Zn_{0.4}Fe₂O₄ Ferrite Magnetic Nanoclusters Using Simple Thermal Decomposition Method. *J. Magn. Magn. Mater.* 2016, 412, 107–113.
- (14) Al-Gaashani, R.; Aïssa, B.; Anower Hossain, M.; Radiman, S. Catalyst-Free Synthesis of ZnO-CuO-ZnFe₂O₄ Nanocomposites by a Rapid One-Step Thermal Decomposition Approach. *Mater. Sci. Semicond. Process.* 2019, 90, 41–49.
- (15) Lv, W.; Liu, B.; Luo, Z.; Ren, X.; Zhang, P. XRD Studies on the Nanosized Copper Ferrite Powders Synthesized by Sonochemical Method. *J. Alloys Compd.* 2008, 465, 261–264.
- (16) Mir, N.; Salavati-Niasari, M.; Davar, F. Preparation of ZnO Nanoflowers and Zn Glycerolate Nanoplates Using Inorganic Precursors via a Convenient Route and Application in Dye Sensitized Solar Cells. *Chem. Eng. J.* 2012, 181–182, 779–789.
- (17) Gholami, T.; Salavati-Niasari, M.; Bazarganipour, M.; Noori, E. Synthesis and Characterization of Spherical Silica Nanoparticles by Modified Stöber Process Assisted by Organic Ligand. *Superlattices Microstruct.* 2013, 61, 33–41.
- (18) Zinatloo-Ajabshir, S.; Salavati-Niasari, M. Facile Route to Synthesize Zirconium Dioxide (ZrO₂) Nanostructures: Structural, Optical and Photocatalytic Studies. *J. Mol. Liq.* 2016, 216, 545–551.

- (19) Kalam, A.; Al-Sehemi, A. G.; Assiri, M.; Du, G.; Ahmad, T.; Ahmad, I.; Pannipara, M. Modified Solvothermal Synthesis of Cobalt Ferrite (CoFe₂O₄) Magnetic Nanoparticles Photocatalysts for Degradation of Methylene Blue with H₂O₂ /Visible Light. *Results Phys.* 2018, 8, 1046–1053.
- (20) Kurian, J.; Mathew, M. J. Structural, Optical and Magnetic Studies of CuFe₂O₄, MgFe₂O₄ and ZnFe₂O₄ Nanoparticles Prepared by Hydrothermal/Solvothermal Method. *J. Magn. Mater.* 2018, 451, 121–130.
- (21) Zakiyah, L. B.; Saion, E.; Al-Hada, N. M.; Gharibshahi, E.; Salem, A.; Soltani, N.; Gene, S. Up-Scalable Synthesis of SizeControlled Copper Ferrite Nanocrystals by Thermal Treatment Method. *Mater. Sci. Semicond. Process.* 2015, 40, 564–569.
- (22) Lopez-Ramón, M. V.; Álvarez, M. A.; Moreno-Castilla, C.; Fontecha-Camara, M. A.; Yebra-Rodríguez, A.; Bailón-García, E. Effect of Calcination Temperature of a Copper Ferrite Synthesized by a Sol-Gel Method on Its Structural Characteristics and Performance as Fenton Catalyst to Remove Gallic Acid from Water. *J. Colloid Interface Sci.* 2018, 511, 193–202.
- (23) Zhuravlev, V. A.; Minin, R. V.; Itin, V. I.; Lilenko, I. Y. Structural Parameters and Magnetic Properties of Copper Ferrite Nanopowders Obtained by the Sol-Gel Combustion. *J. Alloys Compd.* 2017, 692, 705–712.
- (24) Satheeshkumar, M. K.; Ranjith Kumar, E.; Srinivas, C.; Prasad, G.; Meena, S. S.; Pradeep, I.; Suriyanarayanan, N.; Sastry, D. L. Structural and Magnetic Properties of CuFe₂O₄ Ferrite Nanoparticles Synthesized by Cow Urine Assisted Combustion Method. *J. Magn. Mater.* 2019, 484, 120–125.
- (25) Satheeshkumar, M. K.; Ranjith Kumar, E.; Srinivas, C.; Suriyanarayanan, N.; Deepty, M.; Prajapat, C. L.; Rao, T. V. C.; Sastry, D. L. Study of Structural, Morphological and Magnetic Properties of Ag Substituted Cobalt Ferrite Nanoparticles Prepared by Honey Assisted Combustion Method and Evaluation of Their Antibacterial Activity. *J. Magn. Mater.* 2019, 469, 691–697.
- (26) Zinatloo-Ajabshir, S.; Morassaei, M. S.; Salavati-Niasari, M. Facile Fabrication of Dy₂Sn₂O₇-SnO₂ Nanocomposites as an Effective Photocatalyst for Degradation and Removal of Organic Contaminants. *J. Colloid Interface Sci.* 2017, 497, 298–308.
- (27) Masunga, N.; Kelebogile Mmelesi, O.; Kefeni, K. K.; Mamba, B. B. Recent Advances in Copper Ferrite Nanoparticles and Nanocomposites Synthesis, Magnetic Properties and Application in Water Treatment: Review. *J. Environ. Chem. Eng.* 2019, 7, No. 103179.
- (28) Leardi, R. *Experimental Design in Chemistry: A Tutorial.* *Anal. Chim. Acta* 2009, 652, 161–172.
- (29) Mahapatra, C.; Kim, H.-W.; Alqaysi, M.; Han, C.-M.; Singh, R. K.; Owens, G. J.; Foroutan, F.; Knowles, J. C. Sol–Gel Based Materials for Biomedical Applications. *Prog. Mater. Sci.* 2016, 77, 1–79
- (30) Sandesh Jaybhaye, Prajakta Pawar, Bhagyashree Pathai, Shrutika Jaybhaye, Dashrath Munde,

Biosynthesis of Zinc Oxide Nanoparticles for Dye Degradation, International Research Mirror, January 2021, Volume 1, Issue,1, 142-146, ISSN -E 2320-544X, ISSN -P 2250-253X IF- 6.7 7(SJIF).

(31) A. Singh, B. Gaud, Sandesh Jaybhaye, *Optimization of Synthesis Parameters of Silver Nanoparticles and Its Antimicrobial Activity*, Materials Science for Energy Technologies (2020), Vol. 3, 2020, Pages 232-236, doi: <https://doi.org/10.1016/j.mset.2019.08.004> ISSN: 2589-2991 IF 5.04.

(33) Nikhil Pansare and Sandesh Jaybhaye, *Zinc oxide Nanoparticles using hibiscus LeafExtract*, Journal of Emerging Technologies and Innovative Research (JETIR) May 2019, Volume 6, Issue 5, 202-205 (ISSN-2349-5162) [IF 5.87] UGC Approved Journal no 63975.

