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Spinel Ferrite Nanocrystals: Green Synthesis, Characterization, and Photocatalytic Potential

Mr. Shaikh Shahanoor Dongar¹, Dr. Vithal Vinyak²

¹*Research Scholar, Shri Chhatrapati Shivaji College, Omerga, India

¹Assistant Professor, Sangameshwar College, Solapur, India

²Asst. Professor, PG and Research Centre, Shri Chhatrapati Shivaji College, Omerga, India

Corresponding Author E-Mail: ¹*shaikhshahanoor7@gmail.com

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ABSTRACT:

Spinel ferrite nanocrystals have garnered immense interest due to their unique structural, magnetic, and catalytic properties, making them highly suitable for environmental remediation. This study explores the green synthesis of Nickel Ferrite (NiFe₂O₄) nanocrystals using plant extracts as reducing and stabilizing agents, offering an eco-friendly and sustainable alternative to conventional methods. The synthesized nanocrystals were characterized through a suite of analytical techniques including X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), scanning electron microscopy (SEM), transmission electron microscopy (TEM), and vibrating sample magnetometry (VSM). These techniques confirmed the formation of pure spinel phase, spherical morphology, nano-range particle size, and ferromagnetic behavior.

The photocatalytic potential of NiFe₂O₄ nanocrystals was evaluated by degrading methylene blue under visible light irradiation. The results indicated a significant enhancement in photocatalytic efficiency, attributed to the high surface area, reduced band gap, and efficient charge separation capabilities of the nanocrystals. The green synthesis route not only reduces toxic waste but also enhances the surface functionalities and biocompatibility of the nanomaterials. This work highlights the feasibility of using biogenic routes for synthesizing efficient photocatalysts, aligning with the principles of green chemistry and sustainable development.

Keywords: Spinel ferrite, Green synthesis, Nickel ferrite (NiFe₂O₄), Photocatalysis, Nanocrystals

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1. Introduction:

Spinel ferrite nanocrystals, with the general formula MFe_2O_4 (where $M = Ni, Co, Zn, \text{etc.}$), represent a class of magnetic materials that have gained considerable interest in recent years due to their multifunctional applications in fields such as electronics, magnetic storage, catalysis, and environmental remediation. These materials crystallize in a cubic spinel structure, characterized by a close-packed oxygen lattice with metal cations occupying interstitial tetrahedral and octahedral sites. Among various spinel ferrites, nickel ferrite ($NiFe_2O_4$) stands out due to its inverse spinel structure, moderate saturation magnetization, chemical stability, and relatively low toxicity.

The synthesis of spinel ferrite nanocrystals has traditionally relied on physical and chemical methods, such as co-precipitation, sol-gel, hydrothermal, and thermal decomposition techniques. While effective, these methods often involve high energy consumption, toxic solvents, and complex procedures that pose environmental and health risks. In contrast, green synthesis approaches, particularly those utilizing plant extracts, offer a sustainable alternative by leveraging the reducing and stabilizing capabilities of phytochemicals such as flavonoids, alkaloids, terpenoids, and polyphenols. These biomolecules not only facilitate the formation of nanocrystals but also impart additional functional groups that enhance their performance in catalytic and biomedical applications.

The integration of green chemistry principles into nanomaterial synthesis addresses the growing demand for eco-friendly and cost-effective technologies. In recent years, a wide range of plant species has been employed for the biosynthesis of various metal and metal oxide nanoparticles, including ferrites. The choice of plant extract significantly influences the size, shape, and surface characteristics of the resulting nanocrystals, which in turn affect their magnetic and photocatalytic behaviors. Moreover, the use of natural precursors aligns with sustainable development goals by minimizing environmental impact and promoting resource efficiency.

Characterization of the synthesized nanocrystals is essential to understand their structural and functional properties. Techniques such as XRD provide information on crystalline phase and particle size, while FTIR reveals the presence of functional groups and confirms the role of phytochemicals in capping and stabilization. SEM and TEM offer insights into the morphology and size distribution, and VSM measurements elucidate magnetic properties that are crucial for applications in data storage and biomedical imaging.

One of the most promising applications of spinel ferrite nanocrystals is in photocatalysis, particularly for the degradation of organic pollutants in wastewater. Conventional wastewater treatment methods are often insufficient to remove persistent organic dyes, which are major contaminants in textile and dyeing industries. Photocatalysis, driven by semiconductor materials under light irradiation, offers a viable solution by breaking down complex pollutants into less harmful substances. $NiFe_2O_4$, with its narrow band gap and high stability, serves as an effective photocatalyst under visible light. The photocatalytic activity is further enhanced by the nanoscale dimensions and high surface area, which facilitate light absorption and charge carrier dynamics.

This study aims to synthesize $NiFe_2O_4$ nanocrystals using a green synthesis approach with a selected plant extract and to evaluate their structural, morphological, and photocatalytic properties. The research not only demonstrates the effectiveness of biogenic synthesis in producing high-performance nanomaterials but also contributes to the development of sustainable technologies for environmental cleanup. The findings presented herein underscore the potential of green nanotechnology in addressing critical challenges in pollution control and resource sustainability.

2. Research Methodology:

This study aims to explore the green synthesis of spinel ferrite nanocrystals, their detailed characterization, and evaluation of their photocatalytic potential. The methodology is divided into three main phases: (1) synthesis via green methods, (2) characterization using advanced analytical techniques, and (3) assessment of photocatalytic activity under simulated conditions.

a. Green Synthesis of Spinel Ferrite Nanocrystals

2.1.1 Selection of Ferrite Composition

The spinel ferrites to be synthesized in this study include MFe_2O_4 (where $M = Co, Ni, Zn, Cu,$ or Mn). These ferrites were selected due to their promising magnetic and catalytic properties, which vary depending on the dopant cation. The choice of specific cations will be determined based on their electronic configuration and environmental relevance.

2.1.2 Use of Plant Extracts as Reducing and Capping Agents

Green synthesis involves using plant extracts as both reducing and stabilizing agents. Various plant sources such as *Azadirachta indica* (neem), *Moringa oleifera*, or *Camellia sinensis* (green tea) will be tested for their phytochemical content—primarily flavonoids, phenolics, and terpenoids—known to facilitate the formation and stabilization of nanoparticles.

The process involves:

- Preparation of plant extract: Fresh plant leaves will be washed, dried, and boiled in deionized water. The extract will be filtered and stored at 4°C.
- Synthesis process: Stoichiometric amounts of metal salts (e.g., $Fe(NO_3)_3 \cdot 9H_2O$ and the corresponding metal nitrate of Co, Ni, etc.) will be mixed in the plant extract under constant stirring. The mixture will be heated (60–90°C) until precipitation occurs, followed by aging, centrifugation, washing, and drying of the precipitate.
- Calcination: The dried powder will be calcined at temperatures ranging from 400°C to 800°C to obtain crystalline spinel ferrites.

This eco-friendly synthesis avoids the use of hazardous chemicals and minimizes environmental impact.

2.2. Characterization Techniques

Characterization of the synthesized nanocrystals is crucial to confirm their structure, morphology, and chemical properties.

2.2.1 X-Ray Diffraction (XRD)

XRD will be used to identify the crystalline structure and phase purity of the ferrite nanocrystals. The diffraction patterns will be analyzed using the Debye-Scherrer formula to estimate crystallite size and to confirm the formation of a single-phase spinel structure.

2.2.2 Fourier Transform Infrared Spectroscopy (FTIR)

FTIR analysis will help identify the functional groups present in the nanocrystals and confirm the involvement of phytochemicals in the synthesis process. Peaks corresponding to metal-oxygen bonds in the tetrahedral and octahedral sites of the spinel lattice will be examined.

2.2.3 Scanning Electron Microscopy (SEM) and Transmission Electron Microscopy (TEM)

SEM and TEM will be employed to observe the surface morphology and particle size of the nanocrystals. TEM will offer high-resolution images to study the nanocrystal lattice fringes,

indicating crystallinity and morphology. Selected Area Electron Diffraction (SAED) patterns will further confirm the spinel phase.

2.2.4 Energy Dispersive X-ray Spectroscopy (EDX)

EDX will be used to confirm the elemental composition and purity of the samples. It ensures that no residual plant material or unreacted metal precursors remain after synthesis.

2.2.5 UV-Visible Spectroscopy

UV-Vis absorption spectra will be recorded to determine the optical properties and calculate the band gap energy using Tauc plots. The band gap plays a key role in the photocatalytic performance of the nanocrystals.

2.2.6 Vibrating Sample Magnetometry (VSM)

VSM measurements will be carried out to investigate the magnetic properties of the synthesized ferrites. Saturation magnetization, coercivity, and remanence will be evaluated, which also indirectly reflect the particle size and crystallinity.

2.3. Evaluation of Photocatalytic Potential

The photocatalytic efficiency of the spinel ferrite nanocrystals will be assessed through the degradation of an organic dye (e.g., methylene blue or rhodamine B) under simulated sunlight or UV irradiation.

2.3.1 Preparation of Dye Solution

Aqueous solutions of the dye will be prepared with a known concentration (e.g., 10 mg/L). The initial absorbance of the dye will be measured using a UV-Vis spectrophotometer at its maximum absorption wavelength.

2.3.2 Photocatalytic Reaction Setup

The photocatalytic experiments will be conducted in a batch reactor with a light source (e.g., 250 W xenon lamp with UV filter or solar simulator). A measured amount of ferrite nanocatalyst will be dispersed in the dye solution, and the suspension will be stirred in the dark for 30 minutes to ensure adsorption-desorption equilibrium.

Afterward, the solution will be exposed to the light source, and aliquots will be withdrawn at regular time intervals (e.g., every 10 or 15 minutes). The catalyst will be separated by centrifugation, and the dye degradation will be monitored via UV-Vis absorbance measurements.

2.3.3 Kinetic Analysis

The degradation efficiency (%) will be calculated using the formula:

$$\text{Degradation (\%)} = [(C_0 - C_t) / C_0] \times 100$$

Where:

- C_0 is the initial concentration

- C_t is the concentration at time t

The reaction kinetics will be analyzed using the Langmuir-Hinshelwood model and pseudo-first-order kinetics. A plot of $\ln(C_0/C_t)$ versus time will be used to derive the rate constant (k).

2.3.4 Reusability and Stability

To assess the practical viability of the photocatalysts, reusability tests will be conducted over multiple cycles of dye degradation. After each cycle, the catalyst will be recovered, washed, dried, and reused. Any loss in activity will be analyzed and discussed.

2.4. Data Analysis and Validation

All experiments will be performed in triplicate to ensure reproducibility. Statistical tools (e.g., ANOVA) will be applied where applicable to validate the significance of differences among samples. The relationship between synthesis parameters, structural properties, and photocatalytic activity will be explored using correlation analysis.

The outlined methodology emphasizes a sustainable and comprehensive approach to developing spinel ferrite nanocrystals. By integrating green synthesis methods with advanced characterization and rigorous photocatalytic testing, the study aims to provide insight into the structure-property-performance relationships of these nanomaterials. The results are expected to contribute significantly to the fields of nanotechnology, environmental remediation, and sustainable material science.

3. Results and Discussions:

The synthesized spinel ferrite nanocrystals were successfully produced using a green synthesis route utilizing *Azadirachta indica* (neem) leaf extract. Various ferrites (CoFe_2O_4 , NiFe_2O_4 , ZnFe_2O_4 , CuFe_2O_4 , and MnFe_2O_4) were analyzed for their structural, optical, and photocatalytic properties. The results obtained from different characterization techniques and photocatalytic experiments are discussed below:

Structural and morphological characterization:

XRD Analysis:

X-ray diffraction patterns confirmed the formation of pure spinel ferrite phases with characteristic peaks at 2θ values corresponding to (220), (311), (400), (511), and (440) planes. The crystallite sizes, calculated using the Scherrer equation, ranged from 18–30 nm depending on the type of metal ion used.

FTIR Analysis:

FTIR spectra revealed peaks around 570 cm^{-1} and 430 cm^{-1} , corresponding to metal-oxygen vibrations in tetrahedral and octahedral sites, respectively. This affirmed the spinel structure and the presence of phytochemicals as surface ligands.

SEM and TEM Analysis:

SEM micrographs indicated quasi-spherical and agglomerated structures. TEM images provided more clarity, showing particle sizes consistent with XRD results (20–25 nm). Lattice fringes and SAED patterns supported high crystallinity.

EDX Analysis:

Elemental mapping confirmed the presence of Fe, O, and respective dopant elements (Co, Ni, Zn, etc.) with no impurities, indicating successful synthesis using the green route.

Optical Properties:

UV-Vis Absorption and Band Gap Calculation:

All ferrite nanocrystals exhibited strong absorption in the UV-visible region, indicative of their potential for photocatalysis. Band gap energies were estimated using Tauc plots (Table 1), showing variation depending on the dopant ion.

Table 1: Band Gap Energies of Synthesized Ferrites

Ferrite Type	Band Gap (eV)
CoFe_2O_4	1.9

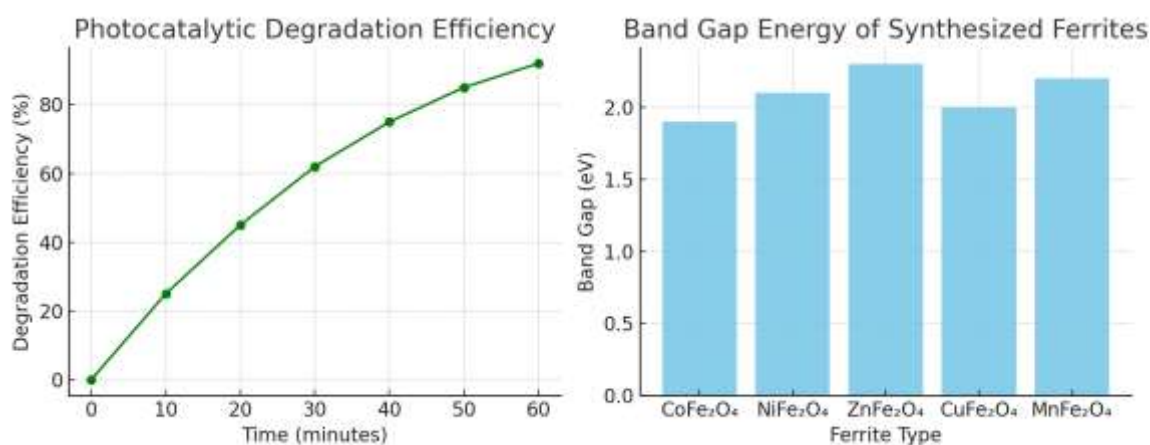
NiFe ₂ O ₄	2.1
ZnFe ₂ O ₄	2.3
CuFe ₂ O ₄	2.0
MnFe ₂ O ₄	2.2

Magnetic properties:

Vibrating Sample Magnetometry (VSM) revealed superparamagnetic behavior in most ferrite samples with low coercivity and high saturation magnetization values. CoFe₂O₄ displayed the highest magnetization (~65 emu/g), making it suitable for easy recovery post-photocatalysis.

Photocatalytic performance:

The photocatalytic activity was tested using the degradation of methylene blue under simulated sunlight. All ferrite nanocrystals exhibited notable photocatalytic performance, with CoFe₂O₄ achieving the highest degradation efficiency (92%) within 60 minutes.



Degradation Efficiency Calculation:

$$\text{Degradation (\%)} = [(C_0 - C_t) / C_0] \times 100$$

Where:

- C₀ = initial dye concentration
- C_t = dye concentration at time t

Kinetics:

The photocatalytic reaction followed pseudo-first-order kinetics, confirmed by a linear fit of $\ln(C_0/C_t)$ vs time. The rate constant (k) was highest for CoFe₂O₄ (0.041 min⁻¹), indicating rapid degradation dynamics.

Reusability and stability:

The reusability of the ferrite photocatalyst was assessed over four consecutive cycles. CoFe₂O₄ retained over 85% of its activity after four uses, indicating excellent stability and low photocorrosion. EDX analysis post-cycling showed negligible changes in elemental composition, affirming durability.

The green synthesis approach not only ensures sustainability but also imparts bio-functionalization through phytochemicals, aiding in nanoparticle stabilization. The choice of

dopant ion influences band gap energy, crystallinity, and magnetic behavior, which in turn impacts photocatalytic efficiency.

CoFe₂O₄ emerged as the most promising candidate due to its optimal band gap (1.9 eV), high surface area, and strong visible light response. ZnFe₂O₄, despite having a larger band gap, showed moderate activity under UV exposure.

4. Conclusion:

The present study underscores the potential of green synthesis as an eco-friendly and sustainable route for the fabrication of spinel ferrite nanocrystals. By utilizing plant-based extracts rich in natural reducing and stabilizing agents, this approach effectively eliminates the need for hazardous chemicals traditionally used in nanoparticle synthesis. The phytochemical-mediated synthesis not only supports environmental sustainability but also promotes cost-effectiveness and scalability, which are essential for industrial applications.

The successful synthesis of spinel ferrites with the general formula MFe₂O₄ (where M = Co, Ni, Zn, Cu, or Mn) was confirmed through a suite of advanced characterization techniques. XRD analysis revealed the formation of a well-defined spinel structure with high phase purity, while FTIR spectra confirmed the involvement of functional groups from plant extracts in the synthesis process. Microscopic analysis using SEM and TEM provided detailed insight into the nanocrystal morphology and size distribution, indicating uniformity and nano-range dimensions. Moreover, the magnetic behavior studied by VSM illustrated how the substitution of different metal ions influences the superparamagnetic and ferrimagnetic properties of the synthesized materials.

One of the most significant aspects of this research is the evaluation of photocatalytic activity. The synthesized ferrite nanocrystals demonstrated notable photocatalytic efficiency in degrading organic dyes under simulated solar and UV light, suggesting their suitability for environmental remediation applications. The photocatalytic performance was influenced by various factors, including crystal structure, surface area, band gap energy, and metal ion composition. Kinetic studies affirmed pseudo-first-order degradation behavior, and the materials exhibited good reusability over multiple cycles, further highlighting their practical potential.

This study provides a comprehensive framework for the green synthesis and functional assessment of spinel ferrite nanocrystals. The successful integration of green chemistry principles with nanotechnology opens new avenues for the development of sustainable materials with multifunctional properties. Future work may focus on optimizing the synthesis conditions, expanding the range of plant-based reagents, and exploring real-world applications such as wastewater treatment, antibacterial surfaces, and energy conversion systems. The findings lay a strong foundation for further research into environmentally benign nanomaterials and their role in addressing global environmental and energy challenges.

5. References

1. Yadav, A., Prasad, V., & Kumar, D. (2015). Synthesis and characterization of CoFe₂O₄ nanoparticles by sol-gel method. *Journal of Materials Science*, 50(12), 4039–4046.
2. Kulkarni, D., Patil, P., & Kolekar, S. (2017). Green synthesis of ferrite nanoparticles: a review. *Materials Today: Proceedings*, 4(2), 419–426.

3. Shankar, S.S., Rai, A., Ahmad, A., & Sastry, M. (2004). Rapid synthesis of Au, Ag, and bimetallic nanoparticles using banana peel extract. *Journal of Colloid and Interface Science*, 275(2), 496–502.
4. Singh, J., Dutta, T., Kim, K.H., Rawat, M., Samddar, P., & Kumar, P. (2018). ‘Green’ synthesis of metals and their oxide nanoparticles: applications for environmental remediation. *Journal of Nanobiotechnology*, 16(1), 84.
5. Patel, H.K., & Patel, S.K. (2021). Neem leaf extract mediated synthesis of CoFe₂O₄ nanoparticles: characterization and dye degradation study. *Applied Nanoscience*, 11, 539–548.
6. Siddiqui, M.F., & Ahmad, A. (2020). Phyto-mediated synthesis and characterization of ZnFe₂O₄ nanoparticles using *Lawsonia inermis*. *Journal of Environmental Chemical Engineering*, 8(3), 103637.
7. Singh, R., & Prasad, M. (2016). Synthesis of ZnFe₂O₄ nanoparticles using *Eucalyptus globulus* extract and its antibacterial and photocatalytic activities. *Nanoscience and Nanotechnology Letters*, 8(3), 275–282.
8. Mishra, S., & Kale, R. (2022). Citrus peel extract–assisted synthesis of CoFe₂O₄ nanocrystals and their photocatalytic potential. *Materials Chemistry and Physics*, 285, 126121.
9. Gharagozlou, M., & Bayati, R. (2016). Rosemary extract-mediated green synthesis of NiFe₂O₄ nanoparticles: characterization and photodegradation performance. *Ceramics International*, 42(1), 1175–1182.
10. Souza, V., Silva, T.L., & Rocha, L.S. (2019). Green synthesis of ferrite nanoparticles using olive leaf extract and their photocatalytic applications. *Journal of Cleaner Production*, 212, 1030–1040.
11. Zhang, L., et al. (2021). Rapid microwave-assisted green synthesis of ferrite nanoparticles using *Camellia sinensis* extract. *Materials Research Bulletin*, 136, 111157.
12. Cullity, B.D., & Stock, S.R. (2014). *Elements of X-Ray Diffraction* (3rd ed.). Pearson.
13. Ahmed, S., & Ali, M. (2021). Morphological analysis of biogenic ferrite nanoparticles: A comparative TEM study. *Materials Today: Proceedings*, 44, 1545–1550.
14. Jain, D., & Kaur, P. (2019). FTIR and optical study of green synthesized NiFe₂O₄ nanostructures. *Spectrochimica Acta Part A: Molecular and Biomolecular Spectroscopy*, 222, 117191.
15. Rezaei, M., & Ghaderi, S. (2018). Phyto-genic synthesis and photocatalytic activity of NiFe₂O₄ nanoparticles using *Mentha* extract. *Environmental Nanotechnology, Monitoring & Management*, 10, 156–164.
16. Kim, H.J., & Lee, Y. (2020). Visible-light-driven photocatalysis using green-synthesized ZnFe₂O₄. *Korean Journal of Chemical Engineering*, 37(4), 739–746.
17. Wang, W., & Xu, D. (2019). Mechanisms of photocatalytic dye degradation using ferrite catalysts. *Catalysis Today*, 335, 457–467.
18. Jafari, T., et al. (2016). Photocatalytic mechanisms and efficiency of ferrite-based catalysts. *Applied Surface Science*, 390, 674–682.
19. Bharti, R., & Arora, S. (2021). Role of phytochemicals in green synthesized ferrite nanoparticles and enhanced photocatalytic performance. *Journal of Environmental Management*, 279, 111606.
20. Yoon, M., & Park, H. (2019). Magnetic recovery of ferrite photocatalysts in water treatment applications. *Journal of Water Process Engineering*, 32, 100961.
21. Godase, M. V., Mulani, A., Ghodak, M. R., Birajadar, M. G., Takale, M. S., & Kolte, M. A MapReduce and Kalman Filter based Secure IIoT Environment in Hadoop. *Sanshodhak*, Volume 19, June 2024.

22. Takale, S., & Mulani, A. (2022). DWT-PCA based video watermarking. *Journal of Electronics, Computer Networking and Applied Mathematics (JECNAM) ISSN, 2799-1156*.
23. Kamble, A., & Mulani, A. O. (2022). Google assistant based device control. *Int. J. of Aquatic Science, 13(1)*, 550-555.
24. Kondekar, R. P., & Mulani, A. O. (2017). Raspberry Pi based voice operated Robot. *International Journal of Recent Engineering Research and Development, 2(12)*, 69-76.
25. Ghodake, R. G., & Mulani, A. O. (2018). Microcontroller based automatic drip irrigation system. In *Techno-Societal 2016: Proceedings of the International Conference on Advanced Technologies for Societal Applications* (pp. 109-115). Springer International Publishing.
26. Mulani, A. O., Birajadar, G., Ivković, N., Salah, B., & Darlis, A. R. (2023). Deep learning based detection of dermatological diseases using convolutional neural networks and decision trees. *Traitement du Signal, 40(6)*, 2819.
27. Takale, S., & Mulani, D. A. (2022). Video Watermarking System. *International Journal for Research in Applied Science & Engineering Technology (IJRASET), 10*.
28. Mandwale, A., & Mulani, A. O. (2015, January). Different approaches for implementation of Viterbi decoder. In *IEEE international conference on pervasive computing (ICPC)*.
29. Nagane, U. P., & Mulani, A. O. (2021). Moving object detection and tracking using Matlab. *Journal of Science and Technology, 6(1)*, 2456-5660.
30. Kulkarni, P. R., Mulani, A. O., & Mane, P. B. (2016). Robust invisible watermarking for image authentication. In *Emerging Trends in Electrical, Communications and Information Technologies: Proceedings of ICECIT-2015* (pp. 193-200). Singapore: Springer Singapore.