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# HIGH-RESOLUTION CHARACTERIZATION OF IRON OXIDE NANOPARTICLES USING TRANSMISSION ELECTRON MICROSCOPY: A DETAILED STUDY

**T B M Chakrapanirao**

Research Scholar, Glocal School of Science

The Glocal University, Mirzapure Pole, Saharanpur (U.P) India.

**Dr. Praveen Kumar**

Research Supervisor, Glocal School of Science, The Glocal University

Mirzapure Pole, Saharanpur (U.P).

**ABSTRACT**

This study explores the characterization of iron oxide nanoparticles and the biodegradability of polymer additive composites. The research focuses on synthesizing iron oxide nanoparticles using the precipitation method, ensuring precise control over particle size and morphology. Various advanced analytical techniques were employed to examine the structural, morphological, and chemical properties of the synthesized nanoparticles. Transmission Electron Microscopy (TEM) analysis revealed that the nanoparticles exhibited sizes of approximately 20 nm, 50 nm, and 100 nm, confirming successful synthesis.Furthermore, the study delves into the integration of these nanoparticles into polymer-based composites, assessing their impact on material properties and biodegradability. The characterization of the composite materials was performed using complementary techniques to understand their physicochemical interactions. The addition of iron oxide nanoparticles influences the degradation behavior of polymer matrices, which is crucial for developing environmentally friendly materials.By evaluating the biodegradability of polymer additive composites, this research contributes to the development of sustainable materials with potential applications in biomedical, environmental, and industrial sectors. The findings highlight the significance of nanoparticle size control and its role in determining the properties of the final composite materials. This study serves as a foundation for future advancements in nanotechnology-driven biodegradable composites.

**Keywords: Photoirradiation, Biodegradation, TEM, Polyolefins, Pro-degradant additive.**

## INTRODUCTION

Polymers are long chain molecules made up of repeating units of monomers. The name is derived from the Greek word *poly*, meaning "many" and *mer*, meaning "part". In other words, polymers are giant molecules of high molecular weight, called macromolecules, which are build up by linking together of a large number of small molecules, called monomers. The reaction by which the monomers combine to form polymer is known as polymerization [1]. The polymerization is a chemical reaction in which two or more substances combine with or without evolution of anything like water, heat or any other solvents to form a molecule of high molecular weight. The product is called polymer and the starting material is called monomer. However recently the emphasis of the biodegradation of polymer has shifted for protecting environment from discarded polymer waste to retrieve value from the used plastics [2]. Biodegradation must be proceeded into two parts biotic and abiotic degradation. As polyolefins are resistant to hydrolysis because they are hydrophobic and not hydro-biodegrade, in presence of antioxidants and stabilizers. Polyolefins are resistant to oxidation and biodegradation, by using pro-degradant additives (promotes oxidation) they can be developing oxo-biodegradable [3,4]. The pro-degradant additives are basically metal salts of carboxylic acids and dithiocarbamates [5,6]. The pro-degradant additives catalyze the breakdown of long molecular chains in polyolefins causes chain separation and production of small molar mass oxidation of products making polymer more hydrophilic [7,8]. Oxo-biodegradation represents two stage process implicates continuous oxidative degradation is also called abiotic degradation followed by biodegradation of the oxidation of products i.e. biotic degradation [9,10]. Nowadays, biodegradability of polypropylene and polyethylene films using pro-oxidant additives are well studied [4,11,12] but to our knowledge biodegradability of PE, PP, EP copolymers in presence of iron stearates and iron diethyldithiocarbamates under accelerated weathering and composting conditions were studied in previous paper no paper on the iron nanoparticles as prooxidant additives.

## MATERIALS AND METHOD

### Material

Commercial polymers such as Polyethylene (PE) [M/s HMA 035], Polypropylene (PP) [M/s Himont USA 70601], Ethylene Propylene copolymers (EPT30U, EPT30R) [M/s Himont Italia], FeCl3.6H2O [Merck] and pro-degradant additives such as Iron stearate (Is), Iron (III) diethyldithiocarbamate (Fd) were obtained from TCI Pharmaceuticals.

### Transmission Electron Microscopy (TEM)

Transmission electron microscopy (TEM) is considered as the most important tool for the high-resolution imaging being useful extensively in extensive scientific fields. TEM gives the direct confirmation about the local structure and the abnormality at the atomic scale of the materials. It is used mostly to resolve the size, shape and arrangement of atoms in the nanomaterials study. Additionally, high resolution transmission electron microscopy (HRTEM) facilitates to know the atomic lattice fringes upto sub-nanometer scale, thus assists to identify the structural arrangement of atoms in nanomaterials . The condenser lens (**Figure 1**) focuses the electrons ray which passes through the object with partial deflection depending upon the sample electron density. The deflection is directly proportional to the mass of the atoms; therefore, the greater the mass of the atoms, greater is the degree of deflection. The sample consisting of low atomic number atoms like C, H, N and O show weak contrasts and needed sample preparations with special contrast enhancing (heavy metals) chemicals . The samples thickness is kept below 100 nm because the increase in temperature and its accumulation due to electron absorption may cause damage to the samples. The scattered electrons passed through sample are collected by an objective lens and the image is formed. The images are further magnified by well-placed supplementary projective lens. The images thus formed are ready to be visible on a fluorescent screen. The images captured with electron microscopes are always in black and white.

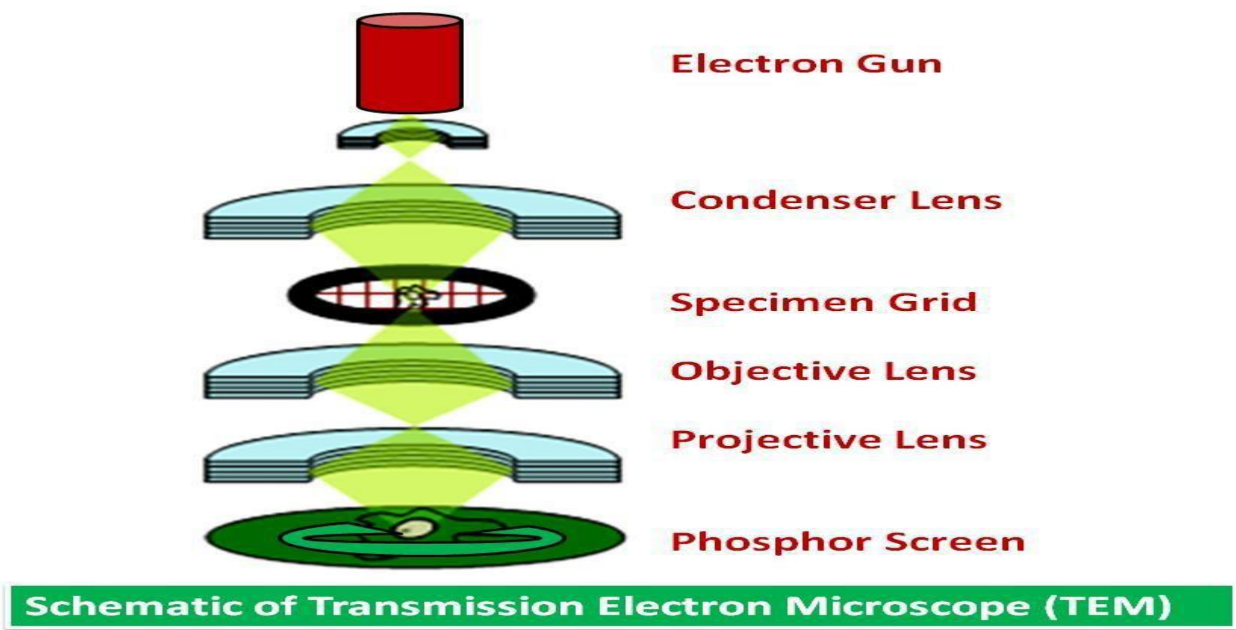



Figure 1: A schematic representation of Transmission electron microscope (TEM)

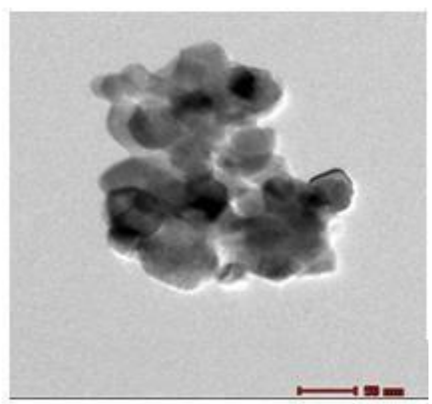
To study higher resolution fine structural details of the polymer nanocomposites transmission electron microscopy (TEM) was employed using a JEOL 1200EX electron microscope operating at an accelerating voltage of 100 kV. The images were captured using a (CCD) charged couple detector camera and processed using Gatan Digital Micrograph software. The polymer samples for TEM imaging were prepared using a Leica

Ultracut UCT microtome at 80-100 nm thickness equipped with a diamond knife at -100 OC. The sections of prepared samples were collected from water on 300 mesh carbon-coated copper grids and dried in vacuum for overnight before loading in the electron microscope chamber.

## RESULT AND DISCUSSION

**Transmission electron microscopy Iron oxide Nanoparticles**

TEM investigations of iron oxide nanoparticles calcined for 4 hr at 500$_0$C figure 5.2 showed the nanostructure which is evident from the TEM. The homogenous distribution and spherical sized particles of ~20,50,100 nanometer is observed.



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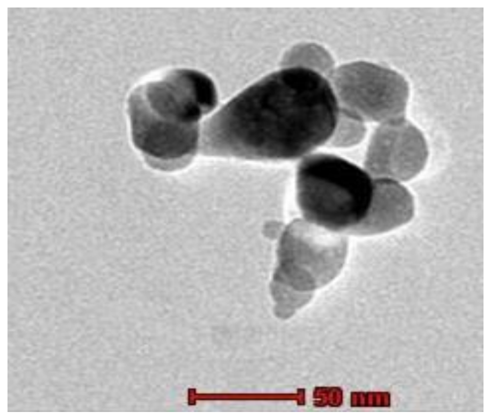


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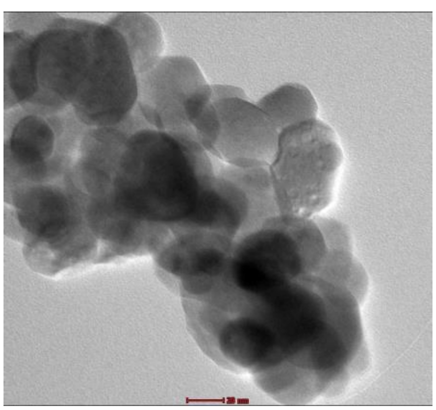

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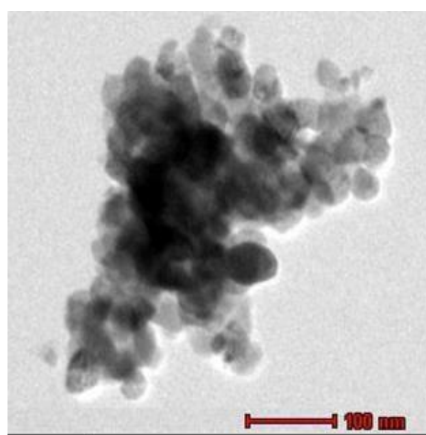


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**Figure: 5.2 TEM micrographs of Iron oxide Nanomaterial a) 20 nm, b) 50nm, c) 50nm, d) 100nm**

The figure showed that the $Fe_2O_3$ formed was discovered - $Fe_2O_3$ has the corundum structure in which each iron atom is surrounded octahedrally by six oxygen atoms, the latter being an hexagonal close packed array.

## CONCLUSIONS

Iron oxide nanoparticles were successfully synthesized using the precipitation method, ensuring controlled formation of nanoparticles with desirable properties. The synthesized nanoparticles were thoroughly analyzed using a range of advanced and complementary characterization techniques to determine their structural, morphological, and chemical properties. Transmission Electron Microscopy (TEM) analysis revealed that the nanoparticles exhibited varying sizes, with observed diameters of approximately 20 nm, 50 nm, and 100 nm. This variation in particle size suggests a degree of tunability in the synthesis process, which can be beneficial for applications requiring specific nanoparticle dimensions. The use of TEM allowed for precise visualization of the particle morphology, confirming their nanoscale nature and uniform distribution. The precipitation method employed in the synthesis process proved to be an effective approach for producing iron oxide nanoparticles with consistent characteristics. These nanoparticles have potential applications in various fields, including biomedical research, drug delivery systems, magnetic resonance imaging (MRI), and environmental remediation. Further investigations involving additional characterization techniques such as X-ray diffraction (XRD), Fourier-transform infrared spectroscopy (FTIR), and dynamic light scattering (DLS) may provide deeper insights into their crystalline structure, surface chemistry, and size distribution. Overall, the successful synthesis and characterization of iron oxide nanoparticles highlight their potential for diverse technological and scientific applications.

**REFERENCES**

[1] Ignacy Jakubowicz (2023) Evolution of degradability of biodegradable polyethylene. Polymer Degradation and Stability 8039–43

[2] Thomas, Clarke, McLauchlin and Patrick (2021) Oxo-degradable plastics: degradation, environmental impact and recyclingWaste and Resource Management 165

[3] Larissa Stieven Montagna, André Luis Catto (2016) Biodegradation of PP films modified with organic pro-degradant:
Natural ageing and biodegradation in soil in respirometric test Polyolefins Journal, Vol. 3 No. 1 59-68

[4] Lucas N, Bienaime C, Belloy C (2008) Polymer biodegradation: Mechanisms and estimation techniques. *Chemosphere*.73(4):429- 442

[5] Ojeda T, Freitas A, Dalmolin E (2009) Abiotic and biotic degradation of oxo-biodegradable foamed polystyrene PolymDegradation and Stability 94(12):2128e33

[6] Alejandro Benítez, Johan J. Sánchez, María L. Arnal, Alejandro J. (2023) Abiotic degradation of LDPE

and LLDPE formulated with a pro-oxidant Additive Polymer Degradation and Stability

[7] Marek Koutny, Jacques Lemaire, A.M. Delort (2006) Biodegradation of Polyehthylene with prooxidant Additives Chemosphere, Elsevier, 64, pp.1243-1252

[8] Chiellini E, Corti A, D'Antone S, Baciu R (2006) Oxo-biodegradable carbon backbone polymers – Oxidative degradation of polyethylene under accelerated test conditions Polymer Degradation Stability 91: 2739-2747

[9] G.J.M. Fechine, D.S. Rosa, M.E. Rezende, N.R. Demarquette (2019) Effect of UV Radiation and Pro-Oxidant on PP Biodegradability Polymer Engineering and Science

[10] Nadras othman, Zulkifli ahmad, Hanafi ismail (2021) Effect of Pro-Degradant Additive on Photo-Oxidative Aging of Polypropylene Film Sains Malaysiana 40, 7: 803–808

[11] David M. Wiles, Gerald Scott (2006) Polyolefins with controlled environmental degradability Polymer Degradation and stability 1581-1592

[12] S. Bonhommea, A. Cuerb, A-M. Delortb, J. Lemairea, M. Sancelmeb, G. Scott (2023) Environmental biodegradation of polyethylene Polymer Degradation and stability 81, 441–452

[13] Sharau Mahadik, Rohini Pawar, Deepti Khanvilkar, Raj Pal Singh (2018) Biodegradability of Polyolefins under Accelerated weathering and Composting conditions IJCAR

[14] J.K.Pandey and R.P.Singh (2021) U.V. irradiated biodegradability of ethylene-propylene copolymers, LDPE, and I-PP in composting and culture environment. Biomacromolecules 2 880.