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STUDY ON CHARECTRIZATION OF IRON OXIDE NANOPARTICLES BY TRANSMISSION ELECTRON MICROSCOPY

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ABSTRACT

This article investigates the Characterization of iron oxide nanoparticles and the biodegradability of polymer additive composites. Iron oxide nano particle were synthesized successfully by precipitation method and characterized using various sophisticated complementary techniques. TEM analysis ~20,50,100 nanometer is observed.

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

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 andnot hydro-biodegrade, in presence of antioxidants and stabilizers. Polyolefins are resistant to oxidation and biodegradation, by usingpro-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 polymermore hydrophilic [7,8]. Oxo-biodegradation represents two stage process implicates continuous oxidative degradation is also calledabiotic 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 [168,169]. The condenser lens (**Figure 4.4**) 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 [168]. 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.

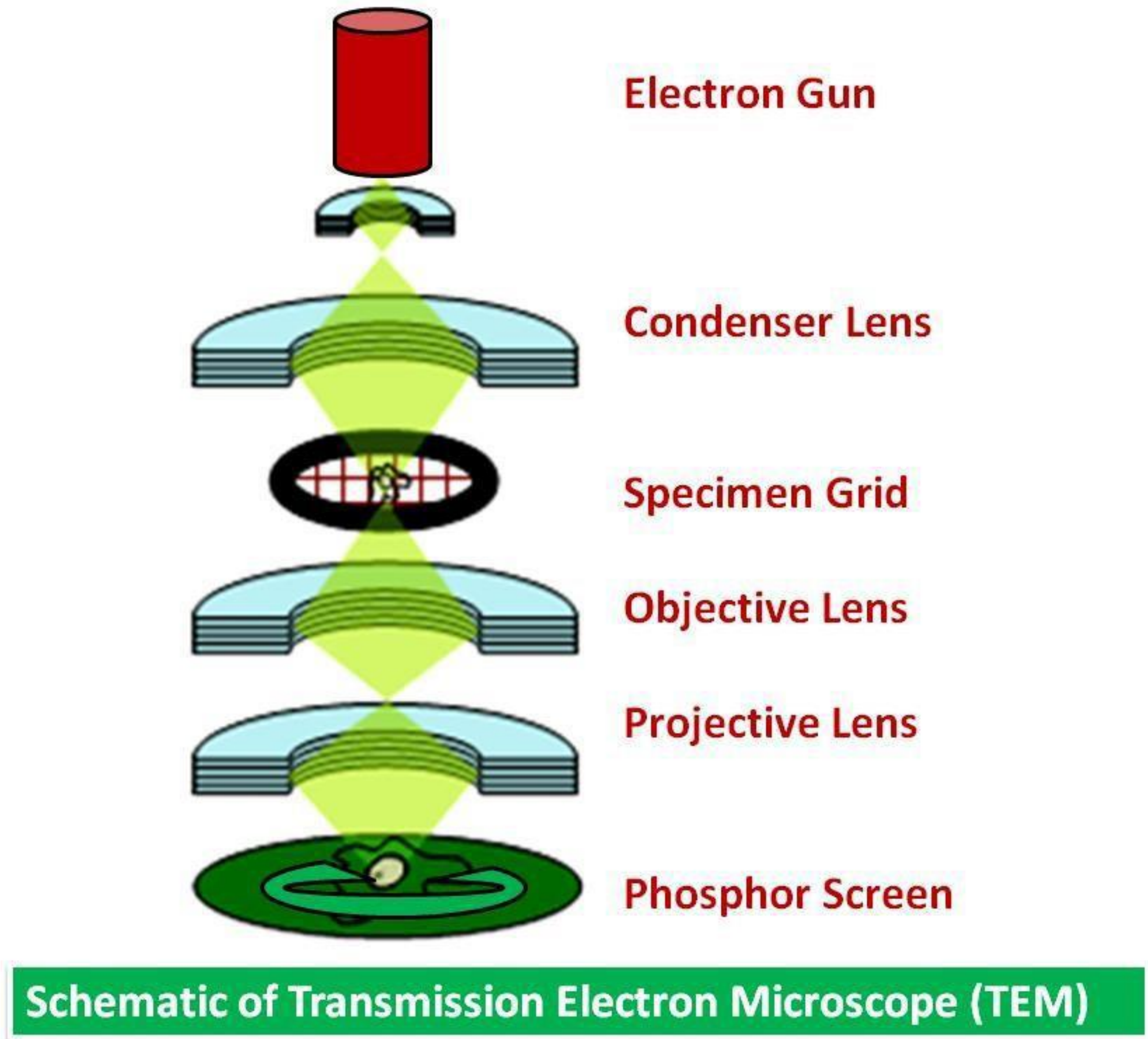


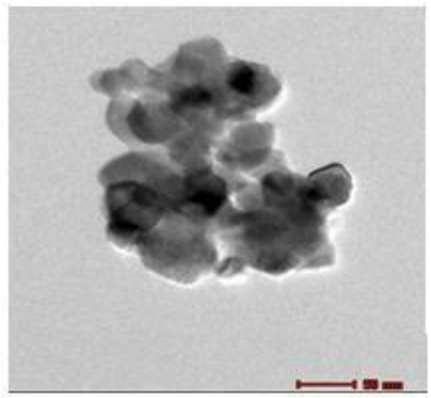
Figure 4.4: A schematic representation of Transmission electron microscope (TEM)

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_0C 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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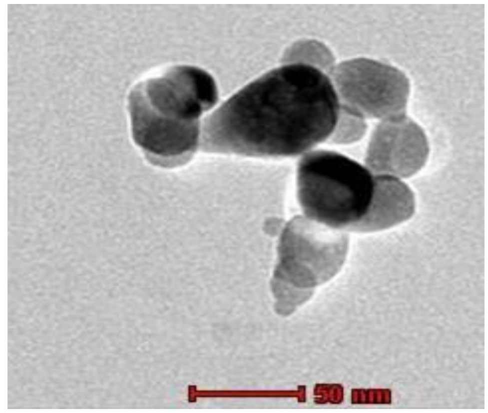

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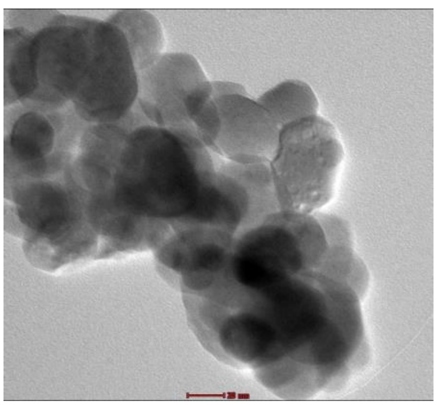

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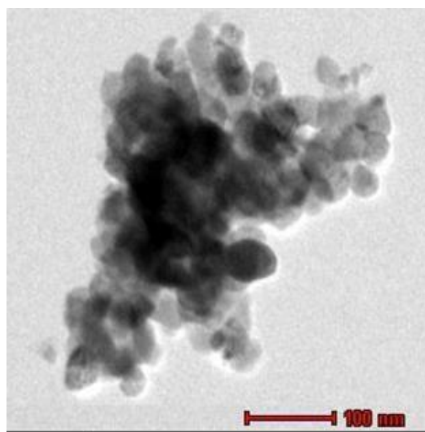

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Figure: 5.2 TEM micrographs of Iron oxide Nanomaterial a) 20 nm, b) 50nm, c) 50nm, d) 100nm

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 nano particle were synthesized successfully by precipitation method and characterized using various sophisticated complementary techniques. TEM analysis ~20,50,100 nanometer is observed.

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