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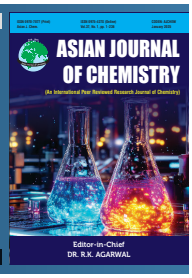

REVIEW

Microplastics Degradation in Water: A Review of Advanced Oxidative Processes and Ozonation for Effective Treatment

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In the backdrop of the increasing microplastics (MPs) production and the global concern into the emergence of pollution, a pressing need to develop extensive implementations of the advanced oxidative processes (AOPs) is in the rise. In line with this, the challenges and competencies of MPs degradation techniques including the identification and quantification technologies used in drinking water and wastewater treatment procedures were analyzed. Assessment of the MPs degradation techniques has shown that photocatalytic processes, Fenton systems and the electrochemical oxidation processes present more challenges than ozone-based technology. Given a much greater solubility of ozone in water that surpasses oxygen, its vigorous reactive properties and the ozone reliable generation, we propose a steady application of ozonation reactions more especially in the disinfection of drinking water, industrial wastewater and river water pharmaceuticals pollution treatment.

Keywords: Pollution, Water treatment, Ozonation, Advanced oxidation processes, Microplastics.

INTRODUCTION

Owing to its outstanding chemical stability, lightweight, long-life span and reasonable cost, its adaptability for numerous applications, plastics industrialization has led to a tremendous comfort to daily human life [1-3]. In 2019, the world's plastics production increased to 368 million tons as compared to 1.5 million tons in 1950. Plastics production is estimated to get to 33 billion tons by 2050 [2,4]. In general plastics is a term used to describe a major family of materials named polymers, large molecules characterized by very large molecular masses and long chain molecular structures. The most common types of plastics include polystyrene (PS), polypropylene (PP), polyethylene (PE), polyvinyl chloride (PVC), polyurethane (PU), polyethylene terephthalate (PET), low-density polyethylene (LDPE) and high-density polyethylene (HDPE). While the plastic products major consumers are ranked from construction, packaging to building industries [4-8].

Plastics are classified conforming to material particle diameter size in the following way: nanoplastics (< 0.001 mm), microplastics (MPs) (0.001-5 mm), mesoplastics (5-50 mm), macroplastics (50-500 mm) and megaplastics (> 500 mm) [5,6,9-11]. Depending on their sources MPs are grouped into two divisions either primary or secondary MPs. Primary MPs include anything from capsules, pellets, fibres, microbeads or nurdles as they are used in table salts, teabags, bottled water, milk, tap water, self-care commodities, cosmetics and in plastic manufacturing processes [7,12,13]. Secondary MPs are formed through the breaking apart of larger plastic portions into tiny ones during prolonged exposure of plastic debris against sunlight and excessive weather [14,15].

Considering inefficient waste management practices and a colossal plastics production, plastics waste is transferred into rivers through sewage and finally reach oceans in the course of time. MPs in wastewater, sewage and the debris that have engulfed the environment have become a plausible threat for the future of water and food security [9,10,16]. Through the food web ingested MPs are transferred through to human food chain [17,18]. As reported through beverage and food a human can ingest between 39,000 and 52,000 MPs particles yearly and in addition around 4,000 to 90,000 MPs particles can be supplemental per year in case tap and bottled water are consu-

med proportionately [10]. Due to their reduced particles size MPs are liable for ingestion by aquatic life. Hu *et al.* [2] reported that 92% out of 690 marine species had ingested MPs debris. As a result of spreading MPs in the ecosystem, wildlife are at greater risk to suffer external and/or internal injuries as well as blockages of the digestive tract [2]. Added to their precarious additives, MPs have an adsorption capacity on their surface for some inorganic and organic toxic pollutants most of which include organochlorine pesticides, dichlorodiphenyltrichloroethane (DDT), polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyls and heavy metals *e.g.* aluminium (Al), silver (Ag), copper (Cu), zinc (Zn) and lead (Pb) [5,8,10]. This exceptional ability makes MPs act as hazardous pollutants collection and distribution vector with a potential of causing harm to humans, wildlife and the entire ecosystem. These threats become more prevalent when environmental condition of temperature, pH, ionic concentration changes. In addition, crystallinity, type and density of polymer can impact on the kinetics of adsorbed contaminants causing desorption and affect physiological and metabolic processes in the eco-system [19-22].

Currently the trend of MPs production, plastic debris have gradually conquered the environment and no stone is left unturned from the northern hemisphere to the southern hemisphere and from the sea depths to the mountain tops threatening the aquatic ecosystem [10,13,18,23]. Traditional wastewater treatment plants (WWTPs) have been rated to be inadequate towards the eradication of MPs. On the perceptible scale MPs are not detected and can effortlessly desert apprehension by WWTPs and eventually integrate the aquatic ecosystem [10,16,23]. Traditional WWTPs fate in the eradication of MPs has been at the fore front of the efforts by engineers and scientists for the establishment of convenient innovative technologies for the degradation of MPs and/or the neutralization of their hazardous effects [6,17,21,24]. Chemical, physical and biological methods have been developed. These methods have nonetheless presented challenges and limitations and are therefore unappealing for an effective removal of MPs from the environment (Table-1) [3,7,10].

TABLE-1
CHALLENGES PRESENTED BY SOME MPS TREATMENT TECHNOLOGIES [Ref. 10]

Technology	Challenges encountered
Membrane filtration	• Membrane clogging • Sludge accumulation • Regular cleaning required
Adsorption, biofiltration	• Elongated adsorption time • Biofilter releases secondary MPs
Coagulation, electrocoagulation	• Regular electrode replacement • Excessive chemical consumption
Magnetic extraction	• Production of secondary MPs • Irrecoverable
Biodegradation	• Elongated degradation time • Production of secondary MPs • Irrecoverable and hard to scale up

To overcome the challenges and limitations presented, the need arose for the scientific community to seek for leading efficient techniques. In this context, advanced oxidation processes (AOPs) are reported as good and harmless methods to the environment and for various wastewater effluents suggested as tertiary treatment considering their resourcefulness and excellent capability to eradicate chemically stable and non-biodegradable contaminants [2,8,12,19,25-27]. Advanced oxidation processes operate on the physico-chemical modifications that yield in molecular fragmentation; the chemical structure simplification followed by a total mineralization. AOPs as an excellent chemical elimination technique involves formation of different reactive oxygen species (ROS) that include hydroxyl ($OH^{\bullet}$), hydroperoxyl ($HO_2^{\bullet}$), superoxide ($O_2^{\bullet-}$), sulphate ($SO_4^{\bullet-}$) and alkoxyl ($RO^{\bullet}$) radicals [5,7,14]. Fig. 1 illustrates a classification of the advanced oxidation processes (AOPs) [28].

Quantification and identification of microplastics (MPs): To identify and quantify MPs, microscopy visual inspection, mass spectrometry and vibrational spectrometry are extensively used techniques. To anticipate the size, shape and colour of MPs visual inspection is the simplest method [29,30], but this way however is subject to bias and will not ensure the chemical composition of microplastics. The chemical composition of MPs can be evaluated through vibrational spectroscopy including Fourier transform infrared spectroscopy (FT-IR) and Raman spectroscopy (RS). For hydrocarbons analysis in general FT-IR is a quick and impeccable technique in use, but detection is only limited to samples ≥ 20 µm [3,4]. To solve this challenge, Raman spectroscopy is used in MPs analysis owing to its detection limit going low up to 1 µm. Xu *et al.* [31] reported a recently evolved surface-enhanced Raman spectroscopy (SERS) with an outstanding ability to detect MPs sizes up to 36 nm. As a technique Raman spectroscopy is however comparably time consuming and expensive [4,16]. The investigation of surface condition will help explain the impact of degradation on the mechanical and physical properties of MPs as the degradation of MPs begins at the outer surface and cautiously drills deeper [13,32].

Scanning electron microscopy (SEM) within digital imaging has been reported by Carraher [33], to provide a larger depth of field as well as higher focus and, is a relevant technique for the evaluation of surface texture, composition and morphology. MPs surfaces have to be coated with conducting materials to get any admissible image. Tofa *et al.* [32] used SEM to evaluate wrinkles, cavities and cracks of different sizes on LDPE films in the establishment of success for the usage of plasmonic enhanced nanocomposite photocatalytic materials made of ZnO nanorods with platinum nanoparticles deposits (ZnO-Pt) to demonstrate the degradation of fragmented MPs under sunlight irradiation. The ZnO-Pt nanocomposite photocatalysts had better degradation kinetics associated with the plasmonic effects giving rise to 78% increase in visible light absorption, enriched hydroxyl radical activity and greater interfacial exciton separation. Liang *et al.* [34] used SEM to exhibit the chalking and surface morphology occurrence of LDPE films after 230 h of UV irradiation. The analyzed image displayed 0.5 to 1 mm deep and 1 to 2 µm wide cavities everywhere on LDPE/TiO_2 film surface and LDPE/MPs-TiO_2.

Transmission electron microscopy (TEM) is one that is a high resolution, all round, useful technique for a variety of

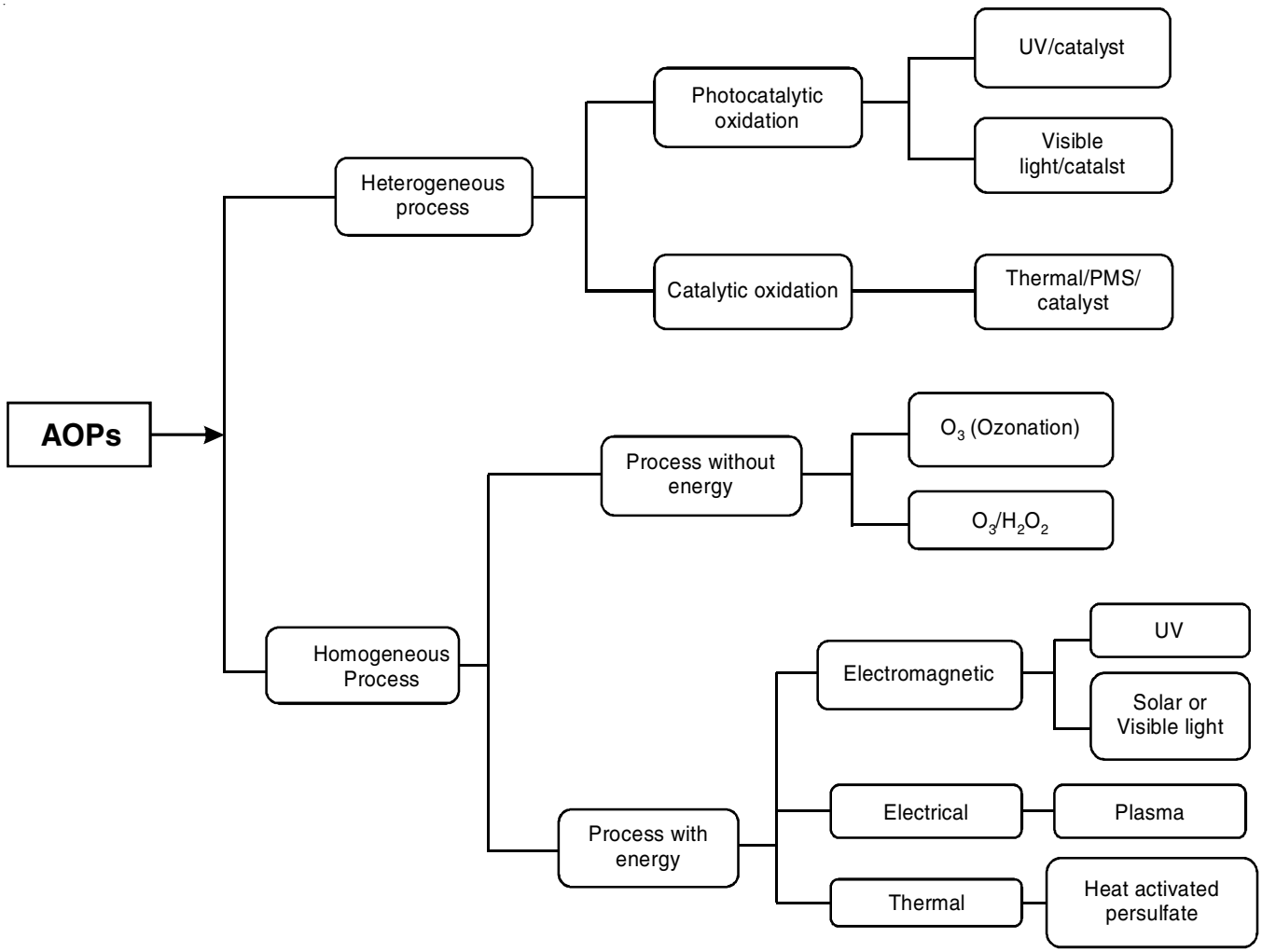

Fig. 1. Presently feasible advanced oxidation processes [Ref. 28]

imaging expertise. It is the one that allows the observation of a detailed structural information at an atomic level, under good working environment magnification can reach up to one million. It provides dark and bright field and is also able to record the samples chemical information, when coupled with a spectrometer the analytical capabilities and imaging improve [33]. Motaung *et al.* [35] investigated the fallout between the amount of titania and the crystalline phase, in the range 1-5 wt.%, on the thermal degradation kinetics, mechanical property and morphology of poly(methyl metacrylate) (PMMA) using TEM and a host of other techniques. The outcome was that titania's structure remained amorphous in the two types of nanoparticles and were well scattered in the polymeric network. Owing to their contrasting carbon composition, size of particle and crystal structures both types of nanoparticles affected the degradation of the polymer in a variety of ways [13,22].

For qualitative and quantitative analysis of MPs, mass spectrometry including pyrolysis coupled with gas chromatography-mass spectrometry (py-GC-MS) are a reliable technique, but the weight limits and upper size are 0.5 mg and 1.5 mm, respectively, curbing its application in complex samples. Huang *et al.* [36] reported similar techniques including thermal depolymerization liquid chromatography-mass spectrometry (LC-MS/MS) and thermal desorption coupled with gas chromatography-mass spectrometry (TDS-GC-MS). Similarly, each technique presents limitations and advantages. Möller *et al.* [37] reviewed other techniques in occurrence liquid chromatography and thermogravimetric analysis (TGA) coupled with differential scanning calorimetry (DSC) or mass spectrometry (MS).

Every plastic material is made of a characteristic chemical composition, a conclusive structure and molecular weight that are prone to changes in a degradation process. Numerous research studies have evaluated MPs molecular weight making use of different techniques such as gel permeation chromatography (GPC), viscosimetry, nuclear magnetic resonance (NMR) and osmometry. These however are only restricted to soluble plastics application. GPC which is also called size exclusion chromatography is an adequate, quick, popular technique that determines a broad spectrum of polydispersity index and molecular weight [10,30].

A high temperature GPC analysis was used to evaluate the variations in molecular weight of LPDE film before and after UV irradiation by Liang *et al.* [34]. They kept the GPC injector and detector temperature at 150 ºC in the analysis. They used an elution rate of 1.00 mL/min with 1,2,4-trichlorobenzene in the mobile phase and reported LDPE composite

reducing in molecular weight as a result of the low molecular weight compounds output.

NMR is an impeccable but comparably expensive spectroscopic technique delivering in both quantitative and qualitative analyses on molecular weights distribution, molecular structure, molecular weights, molecular motion and chain orientation for any microplastic incomparable to other methods [10,22,33]. Izunobi & Giggumbotham [38] observed a solution of *R*-metoxypolyethylene glycol-block-poly-e(benzyloxycarbonyl)-1-lysine dissolved in DMSO-d_6 and R-methoxy-W-aminopolyethylene glycol on a spectrometer at 60 ºC; 499.8 mHz to obtain NMR spectra. They concluded the inability of this technique to evaluate the average molecular weight (Mw) as opposed to GPC technique, its capability in molecular weight calculations is not well appreciated.

On the other hand, viscosimetry and osmometry are comparatively cheaper, less accurate and convenient for calculations on MPs with low and high molecular weight [10,17,33]. Another technique X-ray photoelectron spectroscopy (XPS) has a wide practice for the detection of relative composition, depth profiling and chemical state from the surface of MPs. XPS requires high vacuum conditions, it is comparatively expensive and only caters for surface information. However, XPS provides a complete elemental composition of every MP, unequivocal non-destructive surface characterization of chemical bonding. When exposed to X-rays each element is deemed to elucidate characteristic peaks and relative abundance [10,22,30].

Degradation processes: In the treatment of chemical contamination, advanced oxidation processes (AOPs) have been excessively investigated for wastewater and drinking water resources since their inception in 1980s [14,16,39]. AOPs comprise a combination of various processes utilizing heat, light, plasma and catalysts (Fig. 1). The treatment process successfully produces radicals called reactive oxygen species (ROS). Owing to these radicals high redox potentials, chloride ($Cl^{\bullet}$) (2.0-2.47 V), hydroxyl ($OH^{\bullet}$) (1.89-2.72 V) and sulfate ($SO_4^{\bullet-}$) (2.5-3.1 V), they can promptly diminish a variety of pollutants [13,22,28,40,41].

AOPs are regarded eco-friendly techniques and have been suggested as tertiary treatment for a variety of sewerage discharge given their various applications and advantages: (i) to remove traceable, chemically stable pollutants, refractory non-biodegradable and organic materials, (ii) to raise biodegradability and ensure biological treatment and (iii) a complete conversion of hazardous organic contaminants into inoffensive end products through physico-chemical transformation that leads into the simplification of chemical structure, molecular disintegration and total mineralization [12,28,30].

MPs have become a global rising concern; they have attracted a lot of interest with diverse studies on eradication of MPs being published. At present several review papers on MPs removal through AOPs are reported. Nonetheless, the number of published studies on removal of MPs by ozonation are exceptionally limited. Consequently, it is highly important to recapitulate the different advances of AOPs for the environmental degradation of MPs.

This article is a summarized review of the recent studies on AOPs in general and particularly with an emphasis on ozonation. Fenton processes and photocatalysis are reported to have shown a comparably high degradation efficiency rate positioning them as favourable substitute for the degradation of diverse refractory contaminants such as MPs. But challenges encountered with the industrial application of these techniques during degradation process include amongst others (i) the type and size of MPs, (ii) the light source and its intensity, as well as (iii) the design of the photo reactor [3,8,10,28].

Photocatalytic processes: Photocatalytic process is explained when a light source illuminates a semiconductor (SC) with photons of energy (hν) greater than the energy of its corresponding band gap (E_{bg}), an excited electron migrates to the empty conduction band (C_b) from the valence band (V_b) and an electron pair valence band hole (h_{VB}^+)-conduction band electron (e_{CB}^-) is created at the surface of semiconductor (SC) (eqn. 1). The reactive oxygen species are generated as per reactions eqn. 2, eqn. 3 and, eqn. 4 [28] between the electron pair-hole ($e_{CB}^- - h_{VB}^+$) and reactive species. MPs degradation yields intermediate hydrocarbons that undergo total mineralization into carbon dioxide and water (eqns. 5-6) [16,17,28].

$$\text{Catalyst} + h \longrightarrow h_{VB}^+ \text{ (valence band hole)} + e_{CB}^- \text{ (conduction band electron)} \quad (1)$$

$$e_{CB}^- + O_2 \longrightarrow O_2^{\bullet-} \quad (2)$$

$$h_{VB}^+ + H_2O \longrightarrow OH^{\bullet} \quad (3)$$

$$h_{VB}^+ + OH^{\bullet} \longrightarrow OH^{\bullet} \quad (4)$$

$$OH^{\bullet} + \text{MPs} \longrightarrow \text{Intermediates} \quad (5)$$

$$\text{Intermediates} \longrightarrow CO_2 + H_2O \quad (6)$$

Semiconductors (SC) photocatalysts are classified as single elemental SC such as silicon, tin, germanium, tellurium and selenium. The usage of these single components is reported to have shown limitations associated with low conduction of the charge carrier, low absorption in the visible region and a large number of electron-hole recombination [10,13,30,42].

Double (binary) SC consist of two elements components and are grouped as nitrides (GaN, Ta_3N, g-C_3N_4), meta oxides (ZnO, TiO_2, ZrO_2, CeO), sulfides (CdS, ZnS, CuS) and selenide (CdSe). Ternary SC are three elements components comprising of oxides (ABO_3, AB_2O_4, ABO_2 and ABO_4) with A and B representing metallic cations. Binary and ternary SC as well have a number of drawbacks when used alone. Nitrides or sulfides binary SC have a narrow band gap in addition to instability in aqueous medium, metal oxides exhibit limited reusability, adsorption capacity, higher recombination rates and lower recovery [9,10,13,16,30,31,43].

TiO_2-based semiconductors have been extensively investigated as photocatalysts, however as a result of accelerated rate of recom-bination of the generated electron-hole pairs, a poor quantum yield is exhibited as well as temporary photogenerated carriers and a poorly selective adsorption. Zinc oxide one more metal oxide greatly studied is susceptible to be soluble in both acidic and a saline medium. Some ternary semi-

conductors possess enough narrow electron band gap (E_{bg}) that makes them active when irradiated with visible light, but do not have suitable band edge potential for various reactions [3,22, 31,44-46]. Hamd *et al.* [10] suggested that excellent designs of performing photocatalytic systems can be attained by pairing modified/unmodified semiconductors into nanocomposites or by pairing narrower bandgap binary/ternary semiconductors with larger bandgap binary/ternary ones.

Laboratory prepared TiO_2 film photocatalysts were investigated by Nabi *et al.* [47] for the degradation of polystyrene (PS) and polyethylene (PE) MPs on TiO_2 modified with Triton X-100. They achieved total mineralization at 98.40% efficiency of 400 nm PS in 12 h under UV 365 nm irradiation. By increasing PS size to 5 μm the removal efficacy dropped to 95.3% for 700 nm UV radiation and 93.5% for 1 μm PS after 24 h of UV 365 nm radiation. PS 5 μm was entirely decomposed at 99.9% after 24 h of 254 nm UV light irradiation. PE total mineralization was reached after 23 h of UV irradiation with the principal product being CO_2. It culminates from above results that elevated photons energy, higher electron-hole separation is identified with a greater number of reactive oxygen species (ROS) produced during photocatalytic process will reign in favour of a complete degradation of MPs [13,14,47].

To improve the recombination efficiency of the electron-hole for the removal of MPs, Fadli *et al.* [48] modified TiO_2 photocatalyst with reduced graphene oxide (RGO) and silver (Ag). PE was decomposed at 76% efficiency making use of Ag/TiO_2-1% RGO catalyst within 4 h under UV irradiation while unmodified TiO_2 could only degrade PE to 5% efficiency with the same 4 h time frame. Liu *et al.* [49] investigated PE degradation over time periods 0, 32, 64, 96, 128 and 288 h under 254 nm UV irradiation making use of boron doped cryptomelane (B–OMS–2), they obtained after 288 h of UV illumination a highest removal efficiency of 19.9% in the presence of M-MOMS-2, yet only 9.5% weight loss was noticed without B doping [14,17,30].

The ability of TiO_2/N photocatalyst for HDPE MPs degradation was reported by Ariza-Tarazona *et al.* [50] results showed 1.85% mass loss in the solid environment and 6.4% mass loss in the aqueous environment within 20 h under visible light illumination at room temperature. Ariza-Tarazona *et al.* [51] used the composite C,N-TiO_2 for the degrade HDPE MPs at 0 ºC, pH 3 and obtained a mass loss of 71.77 ± 1.88 % over 50 h. Conversely, Miao *et al.* [52] used a TiO_2/graphite cathode-heterogeneous Fenton system for PVC MPs degradation and achieved 56% weight loss efficiency and 75% dichlorination in 6 h at 100 ºC. Vital-Grappin *et al.* [53] obtained the similar results with Ariza-Tarazona [51] for the HDPE MPs degradation using C,N-TiO_2 powders at a weight loss efficiency of 71.77 1.88%. Llorente-Garcia *et al.* [54] used a mesoporous N-TiO_2 coating for the degradation of HDPE MPs beads and achieved a mass loss of 4.65% within 50 h under visible light 400-800 nm.

Homogeneous Fenton systems: Fenton oxidation processes is one of the prominent and impressive classic category of AOPs due to its favourable capability at ambient state of pressure and temperature, simplicity of procedure, a wide range of applications, interference flexibility, fast degradation and mineralization of the recalcitrant polymers [2,12,52,55,56]. In its fundamental configuration, this system consists of the catalytic reaction of iron ions (Fe^{2+}) with H_2O_2, generating hydroxyl radicals ($OH^{\bullet}$), one of the greatest oxidizing substances with 2.8 V potential *vs.* normal hydrogen electrode [NHE] (eqn. 7) [55,57].

$$Fe^{2+} + H_2O_2 \longrightarrow Fe^{3+} + OH^{\bullet} \quad (7)$$

To ensure continuous production of $OH^{\bullet}$ radicals, the technique requires Fe^{2+} to be reproduced for the entire duration of its oxidation to Fe^{3+}. The rate of reducing Fe^{3+} to Fe^{2+} (eqn. 8) [10] is almost 6000 times lower than the rate of oxidation of Fe^{2+} to Fe^{3+}, it greatly impedes the efficiency of the oxidation/reduction cycle Fe(II)/Fe(III) giving rise to a buildup of Fe^{3+} in the mixture. Fe^{3+} can start to precipitate at pH values above 3 to form iron sludge, a dense contaminant and a drastic decrease of the ferric ions (Fe^{3+}) concentration in the mixture ensures [55,58].

$$Fe^{3+} + H_2O_2 \longrightarrow Fe^{2+} + H^+ + HO_2^{\bullet} \quad (8)$$

Strong acidic medium is a recommendation for the homogeneous Fenton systems. To overcome the drawback caused and spread the pH application of the technique, a number of proposals have been brought forward including firstly the usage of inorganic or organic ligands as chelating agents in the coupling of homogeneous Fenton system with UV-Vis/solar irradiation. Hamd *et al.* [10] reported the usage of nitrilotriacetic acid (NTA), ethylenediamine tetraacetic acid (EDTA), ethylenediamine-N,N′-disuccinic acid (EDDS), oxalate, citrate, tartrate, succinate and carboxymethyl β-cyclodextrin (CMCD) with diverse advantages including an increased yield of $OH^{\bullet}$ radicals and an improvement of Fe^{3+} reduction to Fe^{2+} to generate additional $OH^{\bullet}$ radicals (eqns. 9-10) [57,59].

$$Fe^{III}(C_2O_4)_2^- + h \longrightarrow Fe^{II}(C_2O_4) + C_2O_4^{\bullet -} \ (200\ nm \le \lambda \le 280\ nm) \quad (9)$$

$$Fe^{II}(C_2O_4) + H_2O_2 \longrightarrow Fe^{III}(C_2O_4)^+ + OH^{\bullet} + OH^+ \quad (10)$$

Secondly, the applications of heterogeneous photo-Fenton systems, which have advantages in expanding the pH medium towards a more feasible range given that catalytic reactions are taking place on the surface of a solid catalyst active site. With this there will be a decrease in the leaching of iron ions as well as in the generation of iron sludge, allowing the catalyst reusability. Wang *et al.* [60] studied the usage of Bi_2WO_6/$BiFeO_3$/*g*-C_3N_4 in a heterogeneous photo-Fenton system for an efficient degradation of organic wastes rhodamine (RhB) and tetracycline hydrochloride (TH). They reported an efficient degradation of RhB at 99.85% in 30 min and that of TH at 83.68% in 45 min. Yew *et al.* [61] reported that in a heat activated Fenton system, the thermal energy supply will help increase the upsetting ferric ions (Fe^{3+}) solution temperature causing Fe^{3+} to quickly reduce to Fe^{2+} (eqns. 11-15) [61] averting formation of iron sludge [57,58].

$$Fe^{2+} + H_2O_2 \longrightarrow OH^{\bullet} + OH^- + Fe^{3+} \quad (11)$$

$$OH^{\bullet} + RH \longrightarrow R^{\bullet} + H_2O \quad (12)$$

$$R^{\bullet} + H_2O \longrightarrow ROO^{\bullet} + H_2 \quad (13)$$

$$ROO^{\bullet} + Fe^{3+} \longrightarrow R^{+} + Fe^{2+} + O_2 \quad (14)$$

$$Fe^{2+} + OH^{\bullet} \longrightarrow Fe^{3+} + OH \quad (15)$$

Hu *et al.* [2] studied the efficacy of a hydrothermal coupled Fenton system in strong acid environment for the degradation of polyethylene (PE) and obtained 75.6% mineralization in 12 h and 95.9% weight loss in 16 h. In a comparable study, Lin *et al.* [62] achieved an almost complete degradation of extra-long chain polyethylene into C_3 to C_{20} hydrocarbons at neutral pH with 64% carboxylic acid selectivity through hydrothermal coupled Fenton reactions.

Electrochemical oxidation: Electrochemical oxidation technique as part of AOPs has attained worldwide interest in wastewater treatment. In the electrochemical oxidation process, the driving force is the generation of strong oxidative active species like hydroxyl radical ($OH^{\bullet}$) adsorbed on the electrode's active site (anode) (eqn. 16-19) [63,64].

$$H_2O + M \longrightarrow M(OH^{\bullet}) + H^{+} + e^{-} \quad (16)$$
(M is the electrode material)

$$R + M(OH^{\bullet}) \longrightarrow M + RO + e^{-} \quad (17)$$

$$RO + M(OH^{\bullet}) \longrightarrow M + mCO_2 + nH_2O + H^{+} + e^{-} \quad (18)$$

$$H_2O + M(OH^{\bullet}) \longrightarrow M + O_2 + 3H^{+} + 3e^{-} \quad (19)$$

Recent studies have suggested electrochemical technology as an environmentally safe technique for the degradation of obstinate organic materials as it gives rise to no generation of toxic secondary products [5,65-70]. In this technology, the succe-ssful degradation of MPs can be based on a direct oxidation of the MPs on the anode surface or a direct electron transfer with the help of electric field [68]. The oxidation process relies mainly on the chosen electrode material(s).

In the oxidation reaction, appropriate electrodes best not only undertake a high electrocatalytic exercise for the degradation of MPs in pursuit of desired products but, in turn it should be a low key player for secondary reactions. The electrode specific surface area has been found to be the promoter of transfer and adsorption of the MPs mass on the surface of the electrode [67]. A sizeable quantity of studies has tabled the establishment of innovative electrodes according to noble metals, metal oxides, boron doped diamond (BDD) and dimensionally stable anodes have been launched [65,67-69,71]. Nonetheless, the prospective strategies suffer from challenges of complex synthesis procedures, high cost, release of toxic materials to the environment as well as reduced service lives.

For an effective electrochemical oxidation of MPs, best operating parameters should be determined against current intensity/density, solution pH, electrode plates distance, electrolyte type, anode material, time duration and support electrolyte concentration. Ouarda *et al.* [72] performed an electrochemical oxidation procedure in the treatment of synthetic hospital wastewater against pharmaceutical contaminants such as carbamazepine, ibuprofen, estradiol and venlafaxine. With electrochemical oxidation as post treatment, they achieved a removal efficiency of 97% within 40 min treatment for all four pharmaceutical contaminants at a current intensity of 0.5 A using Nb/BDD as electrodes. A commercial amoxicillin formulation underwent electrochemical oxidation over a BDD in a study by Frontitis *et al.* [73] at a wastewater resource in Patras, Greece. They looked at the effects density of current in the range of 30-50 mA/cm^2, initial pH 3-9, initial COD content of 1-2 g/L, for a treatment time of 15-90 min and concentration of electrolyte 2-4 g/L NaCl. They achieved 78.5% removal efficiency.

Sodium dodecyl sulfate (SDS) was introduced by Lu *et al.* [69] to magnify the degradation performance of polystyrene microplastics (PS MPs) in boron-doped diamond (BDD) anodes. Results showed that SDS addition efficiently improved MPs degradation rate in 72 h of the electrochemical advanced oxidation process (EAOP), around 1.22 to 2.29 times greater than in BDD electrolysis alone. In the same manner, Maharana *et al.* [65] used metal oxide coated Ti anodes including Ti/SnO_2-Sb/Ce-PbO_2, Ti/SnO_2-Sb and Ti/RuO_2 to perform an electrochemical oxidation of 2,4,5-trichlorophenoxyacetic acid. They obtained the degradation efficiency of > 90% within 20 min at initial pH values of between 3 and 11.2 and concentrations ranging between 0.5 to 20 mg/L initial.

The effect of active Ti/Pt or the non-active BDD anodes for electrochemical oxidation of a washing machine effluent was tested by Duran *et al.* [74]. They made use of an applied current of 16.3 and 66.6 mA/cm^2, Na_2SO_4 as support electrolyte at a concentration of 7 g/L. They achieved a total organic carbon (TOC) elimination of > 90% within 360 min with BDD anode at 33.3 and 66.6 mA/cm^2 with the help of SO_4^{2-} and $S_2O_8^{2-}$ that have a weaker oxidizing ability than $OH^{\bullet}$ but possess longer life span. Kiendrebeogo *et al.* [66] achieved a degradation efficiency of 89 ± 8% of electrochemical oxidation of MPs within 6 h on a BDD anode, current intensity of 9 A and density 108.4 mA/cm^2 and a supporting electrolyte Na_2SO_4 at a concentration of 0.03 M. They achieved a total mineralization of MPs at the surface of anode. FT-IR analysis confirmed obtained results with the absence of carbonyl (C=O) bond detections.

Ozone based technology: Ozone is a colourless unsteady gas presenting a suffocating fishy smell under normal atmospheric conditions of pressure and temperature. Ozone is highly soluble in water [75]. The above ozone properties including its solubility, odour and colour each individually have laid a distinct foundation for its broad applications in wastewater and surface water treatment. The exceptional odor is used to signal ozone presence and as a warning for toxic exposure ahead. The widespread ozone applications of in the disinfection of drinking water, wastewater treatment, food preservation and cleaning of air are basically aligned with its energetic chemical properties owed to the strong oxidative potential (2.07 V vis normal hydrogen electrode [NHE]) and its chemical reactivity with a big range selection of inorganic and organic compounds [26,58,76,77].

The ground state of ozone is associated with two sigma (σ) bonds and one pi (π) identified with terminal atoms [75]. Kalemos & Mavridis [78] constructed the ozone resonance structures from its electronic configuration (Fig. 2).

The unsaturated functional groups such as amines, sulfides and other reducing compounds/agents will successfully react

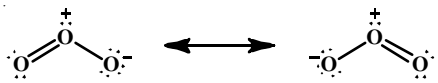

Fig. 2. Ozone resonance structures

with ozone owing to the electrophilic attribute displayed by one oxygen atom having a shortage of electrons as it appears in the ozone resonance structures (Fig. 2). On the other hand, the other oxygen atom has excess electrons displaying nucleophilic property, in favour of reactions with substances containing carbon-nitrogen bonds, carbonyl and strong Brønsted acids [75]. It is evident here that ozone is a vigorous reactive reagent adequate in the oxidation of a large number of contaminants either through direct or indirect ozonation reactions with oxygen containing radicals generated from the decomposition of ozone such as hydroxy radical ($OH^{\bullet}$) with oxidation potential of 2.8 V *vs*. NHE, it is a superb reactive oxidizing agent in the water treatment.

In its conduct, $OH^{\bullet}$ is exceptionally non-selective and readily reacts with diverse organic pollutant species and involved in four reactions *viz*. (i) hydrogen abstraction; (ii) radical addition; (iii) radical combination and (iv) electron transfer [11]. With organic compounds these reactions generate carbon centred radicals $R^{\bullet}$ or $ROH^{\bullet}$, these carbon centred radicals in the presence of O_2 can possibly be modified into peroxyl organic radicals ($ROO^{\bullet}$). A further reaction between all the radicals yields into the generation of more reactive oxidative species (ROS) including hydroperoxide ($HO_2^{\bullet}$), super oxide ($O_2^{\bullet-}$) and H_2O_2. This eventually opens on to chemical degradation as well as mineralization of the target organic compounds.

The direct ozone oxidation is nonetheless a selective reaction in which ozone as a choice reacts to put to use its electrophilic or nucleophilic nature while $OH^{\bullet}$ is generated from ozone decomposition to launch the indirect mechanisms that further accelerate the ozone decomposition process (eqns. 20-31) [75, 79-81].

$$O_3 + OH^- \rightarrow HO_2^{\bullet} + O_2^{\bullet-};\ k_1 = 70\ M^{-1}\ S^{-1} \quad (20)$$

$$HO_2^{\bullet} \rightarrow O_2^{\bullet-} + H^+;\ k_2 = 10^{-4.8}\ M^{-1}\ S^{-1} \quad (21)$$

$$O_3 + O_2^{\bullet-} \rightarrow O_3^{\bullet-} + O_2;\ k_2 = 1.6 \times 10^9\ M^{-1}\ S^{-1} \quad (22)$$

$$O_3 + H^+ \rightarrow HO_3^{\bullet};\ k_3 = 5.2 \times 10^{10},\ k_{-3} = 2.3 \times 10^2\ S^{-1} \quad (23)$$

$$HO_3^{\bullet} \rightarrow OH^{\bullet} + O_2;\ k_4 = 1.1 \times 10^5\ S^{-} \quad (24)$$

$$O_3 + OH^{\bullet} \rightarrow HO_4^{\bullet};\ k_5 = 2.0 \times 10^9\ M^{-1}\ S^{-1} \quad (25)$$

$$HO_4^{\bullet} \rightarrow HO_2^{\bullet} + O_2;\ k_6 = 2.8 \times 10^4\ S^{-1} \quad (26)$$

$$HO_4^{\bullet} + HO_4^{\bullet} \rightarrow H_2O_2 + 2O_3 \quad (27)$$

$$HO_4^{\bullet} + HO_3^{\bullet} \rightarrow H_2O_2 + O_3 + O_2 \quad (28)$$

$$OH^{\bullet} + CO_3^{2-} \rightarrow OH^- + CO_3^{\bullet-};\ k_7 = 4.2 \times 10^8\ M^{-1}\ S^{-1} \quad (29)$$

$$CO_3^{\bullet-} + O_3 \rightarrow (\text{Products})\ CO_2 + O_2^{\bullet-} + O_2 \quad (30)$$

$$OH^{\bullet} + HCO_3^- \rightarrow OH^- + HCO_3^{\bullet};\ k_8 = 1.5 \times 10^7\ M^{-1}\ S^{-1} \quad (31)$$

Decomposition of ozone in aqueous media occurs as a chain process characterized by basic reactions derived from the prominent models by many workers [58,79,82-84]. This decomposition process is inclusive of three main steps: initiation (eqn. 20), propagation (eqns. 21-26) and break in chain reaction steps (eqns. 29-30) as indicated above with k representing the ionization constant. Central to the decomposition process is the free radical initiating step. Instability of ozone molecule is emphasized with the formation of super oxide radical ($O_2^{\bullet-}$) (eqn. 21) or hydroperoxide ($HO_2^{\bullet}$) from hydroxide radical (eqns. 20 and 25-26). In this regard, every species able to utilize hydroxide radicals without re-establishing the super oxide radical ion will create stability of ozone molecule in water [82,83]. Ozone remains reactive despite in liquid or gaseous phase.

Numerous chemical compounds have been found adequate to promote, inhibit, or initiate the chain reaction process [79]. From ozone molecule, the compounds with the capacity to cause the formation of the super oxide ion (O_2^-) are called initiators, such agents are amongst others inorganic compounds [hydroperoxide ions (HO_2^-), hydroxyl ion (OH^-)], organic compounds (formic acid, glyoxylic acid) and humic substances (HS), as well as a number of cations and UV radiations at 254 nm). All inorganic and organic compounds with the capacity to generate super oxide ions from hydroxide radical, which promotes ozone decomposition make the promoters of free radical. Organic promoters include aryl groups, primary alcohols, formic acid, humic acid and glyoxylic acids. Inorganic promoters include phosphates [79]. Inhibitors are chemical compounds that are able to consume hydroxyl radicals ($OH^{\bullet}$) without reforming the super oxide ions (O_2^-), these include alkyl groups, bicarbonate ions and bicarbonates, humic substances (HS) and *tertiary* alcohols (*e.g. t*-butanol) [84-86].

The decomposition of aqueous ozone was studied by Pi *et al.* [87] in the presence of aromatic organic solutes. Aromatic rings were observed to react with ozone or $OH^{\bullet}$ to produce olefins. Ozone reacts with the produced olefin to form H_2O_2, parts of H_2O_2 disband to HO_2^-, which robustly quickens the decomposition of ozone in water.

A study of the ozone-initiated oxidation of cresol isomers was also done by Ncanana & Pullabhotla [80], making use of SiO_2 and γ-Al_2O_3 as adsorbents. They oxidized diverse cresols (*p*-, *o*- and *m*-cresol) over time intervals of 1, 3 and 5 h. They reported that γ-Al_2O_3 adsorbent had a comparatively high catalytic effect for *m*-cresol conversion at 52% efficiency into diethyl maleate (DM), 2,3-dihydroxytoluene (2,3-DT) and *m*-tolyl acetate (*m*-TA). Conversely, SiO_2 advanced the conversion of *o*-cresol at 57% efficiency into *o*-tolyl acetate (*o*-TA), DM and 2,5-dihydroxytoluene (2,5-DT); *p*-cresol at 62% efficiency was converted to *p*-tolyl acetate (*p*-TA), DM and 3,5-dihydroxytoluene (3,5-DT). The ozone-initiated oxidative degradation of saturated hydrocarbons and polymers mechanistic insights was investigated by Lee & Coote [88]. They reported the susceptibility of C–H bond to ozone oxidation to be in the order of *tertiary* → secondary → primary. Amid the polymers studied the order of resistance to C–H bond during ozonation of tested MPs decreases in the order polyvinylchloride (PVC) > poly

methyl metacrylate (PMMA) > polymetacrylate (PMA) > polypropylene (PP) > polystyrene (PS). They also revealed the catalytic effects of water in nurturing C–H bond oxidation process in the polymer systems.

Conclusion

In this review, the potentiality of a variety of techniques to be used to obtain the physico-chemical and thermal properties of microplastics (MPs) including morphology (SEM and TEM); elemental composition (NMR, GPC and XPS), thermal properties (TGA and DSC) are discussed. FT-IR, GC-MS, and LC-MS/MS are the established methods for characterizing the degradation byproducts, which are also investigated. A careful examination of the advanced oxidation processes (AOPs) applications done consisted of photocatalytic processes, Fenton systems, electrochemical oxidation and ozonation process in the refractory organic molecules were investigated. The efficacy of these techniques is focussed around the formation of reactive oxidative species (ROS) such as $OH^{\bullet}$, $O_2^{\bullet-}$ and $HO_2^{\bullet}$ which cause the degradation of polymer chains in MPs. However, it has been observed that photocatalytic processes present some limitations associated with the choice of appropriate semiconductor photocatalysts that will not cause challenges of either low absorption; larger electron-hole recombination; low conduction of the charge carrier; shortened bandgap and limited reusability. Fenton systems present challenges related to a continued supply of Fe^{2+} ions that ensure the production of $OH^{\bullet}$. The rate of reduction of Fe^{3+} to Fe^{2+} is observed to be very low causing precipitation of Fe^{3+} into iron sludge, a dense contaminant that worsens pollution. Electrochemical oxidation processes on the other hand present own challenges related to the choice of appropriate electrodes that will prevent occurrence of adsorption, side reactions and transfer of MPs mass onto the electrode surface. Ozonation is recommended due to its powerful reactivity and solubility in water, which can oxidize a wide range of hydrocarbons directly or indirectly with ROS formed by ozone breakdown. Moreover, ozone has a low potential for the formation of mutagenic products, high disinfecting ability and enhanced biodegradability. Nonetheless ozonation technology is not devoid of challenges. In the initial stage of ozonation in aqueous media, some reactions between ozone and particular dissolved organic matter (DOM) could be in charge of starting off the ozone decomposition and the formation of $OH^{\bullet}$. Omnipresent in DOM, amino, olefinic and phenolic groups readily react with ozone and when deprotonated, they are recorded to partly give rise to $O_3^{\bullet-}$, which immediately decomposes to $OH^{\bullet}$. In order to evolve highly operational and efficient technique with reduced energy expenditure more research should be confined at minimising the radical scavenging effect of DOM and inorganic carbons; synthesis of extremely reactive and ultra-modern ozonation catalysts to expedite ozone decomposition and consequently so the generation of oxygen containing radicals. Catalysts surface condition, chemical composition, electronic properties, morphology, crystal structure should be accounted for during synthesis of choice ozonation catalysts. Special attention needs to be paid at the design of ozonation reactor as well as highly productive ozone generators. High-performance ozone reactor research should emphasize counter current flow design and coordinate fluid dynamics and chemical reaction kinetics.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

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