A brief account of the use of persulfate beyond organic chemistry

Jojiya Grace George, Saima and Joydev K Laha*
Department of Pharmaceutical Technology (Process Chemistry),
National Institute of Pharmaceutical Education and Research
S.A.S. Nagar 160062, Punjab, India
E-mail: jlaha@niper.ac.in

Among various oxidants used for C–C and C–X bond formation in organic chemistry, an inorganic oxidant persulfate [mainly $K_2S_2O_8$, $Na_2S_2O_8$ and $(NH_4)_2S_2O_8$] embraced a prominent position due to its unique efficiency towards bond formation, cost-effectiveness, easy commercial availability, and environment friendly nature. Unlike several reviews available on the use of persulfates in organic synthesis, any specific account addressing the other uses of persulfates is currently unavailable. The current review focused on composing a brief account of the literature that describes various other uses of persulfates in biological and inter-disciplinary sciences followed by drug degradation.

Introduction

In classical chemistry term, we can describe oxidant as a chemical species that possesses the ability to gain one or more electrons, and subsequently oxidizes other substances present in a redox reaction. A vast array of oxidants that have been discovered with a decipherable use in these reactions include molecular oxygen, halogen compounds (hypochlorite, chlorite, perchlorate, chlorate), hexavalent chromium compounds such as chromic and dichromic acids and chromium trioxide, permanganate compounds, cerium compounds (ceric ammonium nitrate and ceric sulfate), $PhI(OAc)_2$, lead dioxide, TBHP, persulfates, etc. Among these varieties of oxidants, potassium persulfate is a rational choice as an oxidising agent with wide applications in anextensive range of laboratory experiments to industrial processes with listed a few.[1]

From the variety of per-oxygen family compounds, the peroxydisulfate ion ($S_2O_8^{2-}$) is known as the most prevailing oxidant and which can generate sulfate radical anion ($SO_4^{\bullet-}$) by following thermal, photolysis, radiolysis, transition metal catalysis, or redox decomposition path under mild conditions. Moreover, it has been already documented in literature and widely known that the sulfate radical anion ($SO_4^{\bullet-}$), which has been generated from any metal persulfate, is furthermore accountable for the strong oxidizing property of the metal persulfate. In addition, many reports already are available about the very strong one-electron oxidant nature of $SO_4^{\bullet-}$ possessing redox potential of 2.5-3.1 V.[2] Moreover, further analysis by various research groups explained its longer lifetime (~4 s) than hydroxyl radicals, largely due to their preference for electron transfer reactions. Though it is already known in literature that an electron transfer process could either be exergonic or endergonic, hitherto, the endergonic process might be slow due to the involvement of higher activation energy in the reaction process. $K_2S_2O_8$ is also known for its oxidizing capability for various metal salts and complexes, anions, nucleophilic radicals, and neutral organic compounds. In the present scenario, main focus is to use or employ various green chemistry principles, and thus use of $K_2S_2O_8$ becomes extensive matter of study. Various reports have appeared in literature leveraging the use of this oxidant in various metal-catalysed and metal-free organic transformations.[3] Moreover, this point needs to be highlighted mainly as $K_2S_2O_8$ is widely explored and extensively used persulfate, comparable to its other variants, such as $Na_2S_2O_8$ and $(NH_4)_2S_2O_8$, the reason behind this might be the good solubility of potassium salt in organic solvent, which further act as an adding point in the reaction transformation.[3] It has further utilized as an effective oxidant in both metal-catalyzed and metal-free reactions for the formation of C–C to C–X (X = N, O, S, P, B, Si, F, Br, I) bond across a broad range of substrates. Apart from employing this oxidant ($K_2S_2O_8$) in a wide array of oxidative organic transformations, recently the progress is seen for this oxidant to use in the field of biology and medical science. Being cheaper and readily availability, $K_2S_2O_8$ is used in a wide cluster of advanced

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oxidation processes (AOPs).[4] The current review is a brief account of the literature that describes various other uses of persulfates in medicinal chemistry, biological and inter-disciplinary sciences.

In 2018, a comprehensive review highlighting the recent advancement of potassium persulfate in oxidative organic transformations involving both metal catalyzed and metal-free reactions, which was contributed by our research group.[3] However, no review is available, which is specifically focused on the various roles of potassium persulfate in the research area other than organic reactions transformation. We believe that this review should fill in as a valuable reference to the scientists in creating noteworthy progress in this research area. We herein listed some of the uses of potassium persulfate in various research area other than organic synthesis.

Use of potassium persulfate in medical sciences

We are well aware about the blood transfusion, is a lifesaving procedure in which blood or blood products are transferred into patient's circulatory system. This intervention is used to tackle the emergencies and life threatening injuries. More importantly the donated blood cannot be considered as safe due to their inherent side effects.[5] However, various sensitive methods are available for the frequent detection of the blood bornediseases existing in donor's blood, risk of exposure to blood borne pathogens like AIDS, HIV could happen in allergenic blood transfusion.[6] Moreover, other complications associated with blood transfusion include agglutination caused due to inaccurate cross matching of different blood classifications, absence of accessibility of donor in specific circumstances, carefully refrigerated surrounding for blood storage and so forth.[7] To combat all these challenges, these limitations can be overcome with the utilization of artificial red blood substitutes.

Thus in this context, in 2015, RuifenXu and co-workers used potassium persulfate for an vital application in the preparation of a novel artificial red blood substitute known as grafted starch-encapsulated haemoglobin. The strategy involved in the preparation of artificial red blood cells, is to encapsulate haemoglobin with long chain fatty acid grafted with potato starch. Now the perception here is the role of potassium persulfate, which is employed as a catalyst for the purpose of grafting starch with fatty acid in DMSO, followed by the encapsulation of haemoglobin onto the grafted starch polymer that in turn resulted in the formation of grafted starch-encapsulated haemoglobin.[8]

Role of potassium persulfate as a disinfectant against microorganisms

Potassium persulfate is considered as new and efficient disinfectant, which is preferably used in livestock, aquaculture and poultry. This oxidant ($K_2S_2O_8$) exhibited higher capacity for removal of aquatic toxic material and has high efficiency in killing microorganisms. It might be possible that the potassium persulfate acts on microorganisms by oxidation, as persulfate is capable of producing $SO_4^{\bullet-}$ as the strong oxidizing agent. The main advantage associated with potassium persulfate is that it didn't affect or damage the human beings, animals and ecological environment, which further broadens the scope of persulfate application comparable to the other classical disinfectants.[9]

In another instance, use of potassium persulfate as an incredible disinfectant against antibiotic resistant bacteria (ARB) and antibiotic resistant genes (ARG) has been explored by Justins and co-workers. An investigation was conducted for improved ARB and ARG removal by comparing various currently used disinfection techniques like chlorination, E-beam, and prospective ozonization techniques involving potassium persulfate in combination with several catalysts. From the experiment, it was concluded that hydrogen peroxide could possibly be substituted with potassium persulfate, as it increased the effectiveness of ozone in disinfecting ARB and ARGs.[10]

Similarly, Janne Lunde and co-workers in 2013 found the application in food industries wherein they demonstrated the use of potassium persulfate as a potent disinfectant for the elimination of *L. monocytogenes* in food processing plants, which otherwise appears to persist despite regular disinfection causing prolonged contamination. It's persistence might be due to the adaptivity of the *L. monocytogenes* for all the disinfectants. The only disinfectant that *L. monocytogenes* did not adapt to was potassium persulfate and the MIC for all the strains was found to be 2500µg/ml.[11]

In 2016, Afonyushkin and co-workers found the application of potassium persulfate against some pathogenic bacteria. They studied the effect of a biocidal product, Ecocid based on potassium persulfate, on the genetic material of pathogenic bacteria specific to meat processing industry. It has been inferred from the results that the concentration of the chromosomal DNA of *Clostridium* perfringes was reduced to 28-29 times due to the tested disinfectant based on potassium persulfate.[12]

In other report, which also documented the application of potassium persulfate for bactericidal activity was given by Ashley and co-workers wherein they contemplated the specific bactericidal action of potassium persulfate, which demonstrated that potassium persulfate behaves as a "physicochemical" germicide preferentially attacking *B. fluorescens non-liquefaciens*. This bactericidal activity of potassium salts might be because of the way that these salts induce physical changes in the proteins as well as exerts their oxidising capacities on the microorganisms.[13]

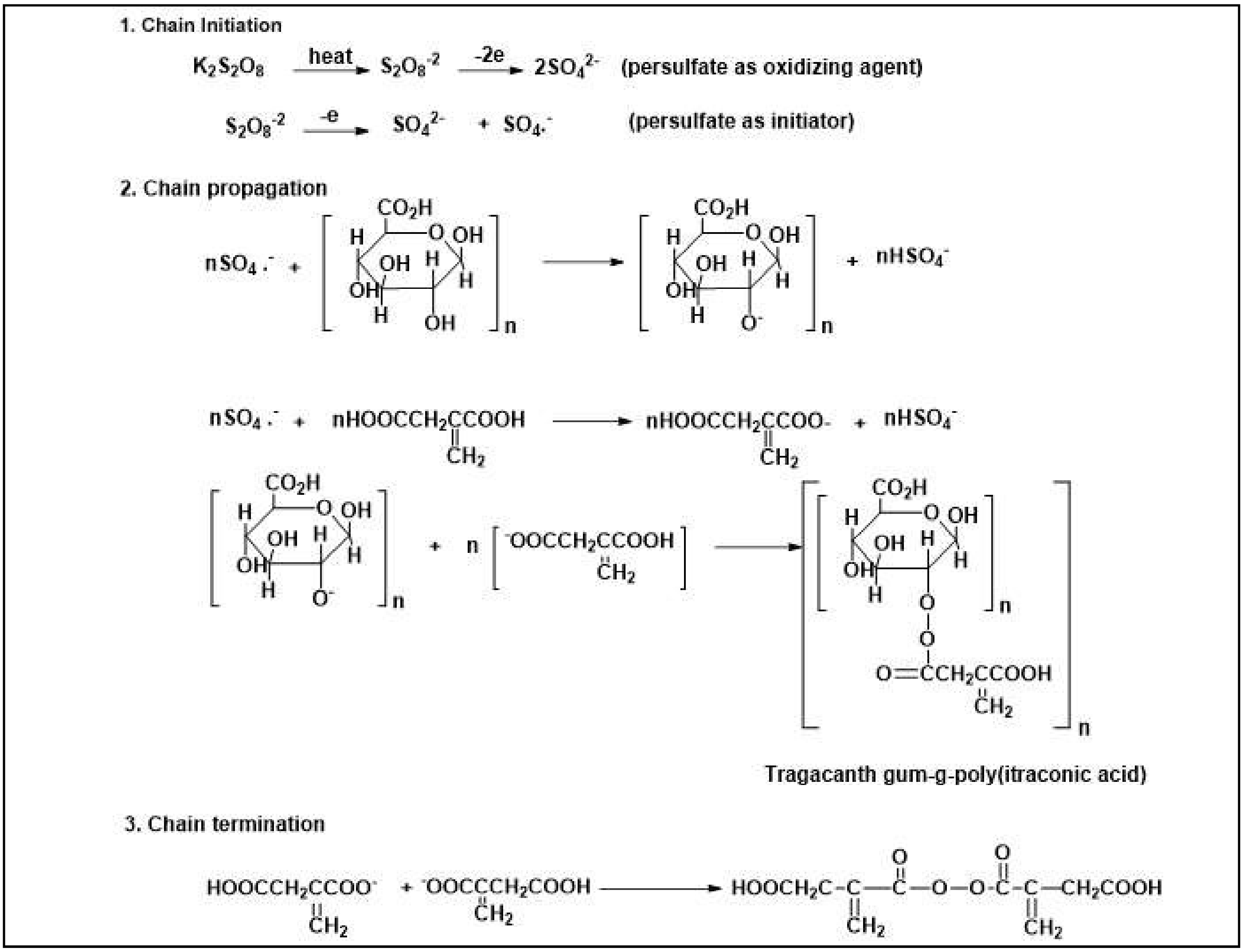
Potassium persulfate as an initiator in polymer chemistry and nanotechnology:

Potassium persulfate being a strong oxidant, has been very nicely employed used to initiate polymerization reactions, which can indeed found application in the polymer making industries for the synthesis of important polymers, beneficial in medicinal chemistry, or for commercial use.

Thus in 2018, Deepak and co-workers used potassium persulfate for grafting wherein potassium persulfate acts as an initiator for grafting of tragacanth gum with itaconic acid in the presence of N,N′-methylene-bis-acrylamide as cross linker. In addition, the synthesised polymer has significant antimicrobial efficacy against E.Coliby agarwell diffusion assay.

Scheme 1nicely demonstrated the mechanism of nanohydrogel formation. The major steps involved in polysaccharide modification, involve the formation of ester and ether linkage with sugar hydroxyl groups. These hydroxyl groups make the reaction site assessable for alterations and cross linkages in tragracanth gum.[14]

Adding another example for polymer synthesis application, in 2017, Sood and co-workers utilized potassium persulfate as a cross linking agent. The group has demonstrated the use of microwave for the preparation of carboxymethyl cellulose-cl-poly (lacti acid-co-itaconic acid) hydrogel in the presenc of potassium persulfate as linking agent. During this preparation process, N,N′-methylene-bis-acrylamide serves as an initiator. The effect of potassium persulfate was studied, which showed that maximum grafting could be obtained at 0.3 g of potassium persulfate. Active sites are generated on the polymeric backbone by the initiator and monomers causing increase in grafting.[15]



Scheme 1. Mechanism of grafting itraconic acid on tragacanth gum.

Another hydrogels formation was demonstared by Guanghua et.al, wherein they prepared super absorbent hydrogels by grafting N-succinyl chitosan with poly (acrylic acid-co-acrylamide). In the preparation process, potassium persulfate and N,N′-methylenebisacrylamide were used as initiator andcross linker, respectively. In the study, the effect of potassium persulfate content on the water absorbency for the super absorbent hydrogel was observed, which showed that there was an increase in the water absorption capacity of hydrogel from 767 to 1375g/g and then decreased to 503g/g. The absorption of PAMAM dendrimer in alkaline or lower concentration solution also inhibits the growth of Escherichia coli, which demonstrates its antibacterial application.[16]

To extend further application of potassium persulfate in the recent technology, various reports are available in literature in the nanoscience wherein its utilization has been employed. Thus, in 2017, Dipankar and co-workers used potassium persulfate as an initiator for the preparation of functionalized dextrin based cross-linked, pH responsive and biocompatible nanogel for the targeted delivery of doxorubicin hydrochloride to human osteocarcoma cancer cell lines. In this report, they used Michael-type addition reaction for the preparation of nanogel using dextrin. The other components involved in the reaction include N,N′-methylene bisacrylamide that functions as a cross linker and acrylic acid that serves as a monomer.[17]

Guanghua et al. synthesized quaternary ammonium chitosan-g-poly(acrylic acid-co-acrylamide) superabsorbent hydrogels from acrylic acid, acrylamide and quaternary ammonium chitosan, wherein potassium persulfate was used as an initiator and N,N′ methylenebisacrylamide as a cross linker, respectively. The effect of potassium persulfate content on the water absorbency of the super absorbent hydrogel was evaluated during the study. They also observed that with a change in the potassium persulfate content from 0.25 to 0.75 %, an increase in water absorbency in distilled water from 292 to 429 g/g was observed and reached the maximum with 0.75 % of the initiator content. Further increase in the initiator content leads to lowering of water absorbency. The antibacterial study indicated that the antibacterial activity was improved on increase in the content of initiator, but the activity would decrease if more initiator is added beyond a certain limit.[18]

In polymer science, another example was given by Rajeev and co-workers in 2016, where they used potassium persulfate as a thermal initiator for the free radical polymerization initiation during the synthesis of poly (acrylamide)-based hydrogel, which serves as the polymer matrix in the preparation of Gum rosin acrylamide copolymer-based nanogel. Formation of GrA-cl-poly(AAm) is facilitated by grafting poly(acrylamide)chains onto the polymeric backbone employing $OH^{\bullet}$ as the most active site. Potassium persulfate is decomposed to form $SO_4^{\bullet -}$, which reacts with water to generate $OH^{\bullet}$ free radicals at an ambient temperature. The polymeric backbone reacts with the $OH^{\bullet}$ free radical and monomer resulting in the formation of active sites. Finally, the active graft polymer is formed by grafting of poly (acrylamide) chains onto this active site of backbone, which in turn undergoes crosslinking with MBA to from cross linked hydrogel having more antibacterial activity against S.aureus as compared to *E.coli*.[19]

In 2015, Bajpai and Mamta employed potassium persulfate as an initiator and N,N′-methylene bisacrylamide as cross-linker for the free radical initiated aqueous polymerization of sodium acrylate monomer for the fabrication of gum acasia/ poly(sodium acrylate)semi-interpenetrating polymer networks(Semi-IPN) that was further used for the in situ preparation of *Syzygiumaromaticum* loaded silver nanoparticles having antibacterial activity against E.coli.[20]

In 2012, Nadia and co-workers used potassium persulfate as an initiator in the grafting of carboxymethyl chitosan with N-acryloyl, N′-cyanoacetohydrazide in homogenous aqueous phase. Nitrogen radicals were reported to be formed on carboxylmethyl chitosan after reacting with potassium persulfate, which upon radical addition to N-acryloyl,N′-cyanoacetohydrazide and subsequent polymerization generated various copolymers. The maximum grafting yield achieved for 0.03 mol/L potassium persulfate was 44.8 % at 60 ºC within 2 h. These grafted polymers showed an inhibitory effect on both *E. coli* and *Staphylococcus aureus* bacteria followed by *Aspergillus favus* and Candida albicans fungi which were better than those of chitosan and carboxymethyl chitosan themselves.[21]

In the same year, Kamel prepared silver-nanoparticle (AgNP) containing paper exhibiting antibacterial activity. Polymerization was carried out by grafting acrylamide onto bagasse paper sheets under microwave conditions using potassium persulfate as an initiator. The loading of silver-nanoparticle on the acrylamide grafted baggase paper sheets occurs by in situ reduction of silver nitrate with citrate molecule as a stabilizing ligand.[22]

In 2011, Anirudhan and Sandeep synthesized and characterized a novel pH-controllable hydrogel by in situ intercalation graft copolymerization of 2-acrylamido-2-methylpropane sulfonic acid (AMPS) with N-malleoylchitosan (MACTS) intercalated montmorillonite to form 2-acrylamido-2-methylpropane sulfonic acid (AMPS) grafted N-malleoylchitosan (MACTS) intercalated montmorillonite for anticancer drug delivery. This graft polymerization was carried out by potassium persulfate as a free radical initiator and N, N-methylenebsacrylamide as a cross-linking agent.[23]

Another example in the nano drug delivery was given in 2011 by Julio and Edward, wherein they developed a convenient method for the preparation of emulsified polyacrylate nanoparticles for the delivery and protection of ß-lactam antibiotics. In this example, potassium persulfate acts as a water-soluble initiator in the emulsion polymerization using butyl acrylate and styrene in water in the ratio 7:3, containing sodium dodecyl sulfate as a surfactant resulting in polyacrylate-based nanoparticles. These emulsions were purified by centrifugation and dialysis, which upon lyophilisation formed a homogenous dried powder, which further can be reconstructed by an aqueous diluent forming a nanoparticle emulsion for the application in the field of drug delivery.[24]

Hydrogels synthesis application for bacterial inhibition was demonstrated by Saxena and Bajpai in 2010, prepared cationic-resin (seralite SRC-120)-loaded poly (acrylamide) gels by free radical polymerization of acryl amide in the presence of resin particles. This reaction was initiated by potassium persulfate and methylene bisacrylamide serves as the cross-linker. The resin loaded hydrogel exhibited minimum swelling property and was found to have greater inhibitory activity against E.coli as compared to silver-nano particle loaded hydrogels.[25]

Huajiang et al. used potassium sulphate as an initiator for the free radical polymerization of an antibacterial quaternary ammonium acrylic monomer, which was synthesized by the quaternization of 2-dimethylamino ethyl methacrylate with dimethylsulfate. The corresponding homo-polymers when tested against E. coli and S. aureus showed significantly stronger antibacterial activity as compared to the monomer. From the study it was concluded that the number of polymer molecules were increased directly with increasing the potassium persulfate concentration, while the degree of polymerization was decreased which on the other hand indirectly lead to a decrease in the viscosity.[26]

In 2008, Loannis and co-workers found application in the emulsion formation and thus employed potassium persulfate as an initiator for the emulsion polymerization of styrene to form anionic polystyrene lattices that was used in the synthesis of hollow ceria nanospheres. Radicals are penetrated into the micelles and the molecules present in the monomer will diffuse continually from the monomer spheres to the micelles through the solvent until all the monomers are eliminated to form the polymers.[27]

In 2007, Sampath and co-workers prepared glycosylated polyacrylate nanoparticle from glycosylated drug monomers by the emulsion polymerization technique in which potassium persulfate was utilized as a radical initiator. The emulsion polymerization was carried out using a mixture of butylacrylate-styrene in 7:3 ratio (w:w) in the presence of sodium dodecyl sulfate as surfactant along with potassium persulfate which served as a radical initiator. The average diameter of the glyconano particles was about 40 nm by dynamic light scattering (DLS). The resulting nanoparticles possessed equivalent or improved in-vitro antibacterial activity as compared to their precursor monomers.[28]

Bajpai and co-workers found use of potassium persulfate in wound dressing and thus applied it as an initiator along with the cross linker, N,N'-methylene-bis acrylamide in soaking plain cotton cellulose (CC) fibres in a reaction mixture containing monomer acrylic acid to produce cotton cellulose/ polyacrylic composite fibres. These fibres could be utilized to release the entrapped drug Gentamicin sulfate in the physiological fluid for burn/wound dressing applications. These fibres when tested demonstrated significant mechanical strength and fair biocidal activity against *E. coli*.[29]

Fahreia and co-workers grafted N-acryloylmorpholine (NAM) onto chitosan by utilizing 1% acetic acid as the solvent under homogenous conditions, wherein potassium persulfate and sodium sulfite were used as the redox initiator. The grafted polymer remarkably demonstrated an improvement in crystallinity along with a stronger inhibitory activity on bacteria and fungi as compared to chitosan.[30]

In 2013, Wenning and co-workers designed potassium persulfate as an oxidant for the preparation of divalent silver oxide-diatomite (AgO-d) hybrids by chemical oxidation using silver nitrate and diatomite as the raw materials. AgO-d hybrids when tested for antibacterial activity showed excellent bactericidal activity against *S. aureus* and *E. coli.*[31]

In 2013, Fakhreia et.al employed potassium persulfate in combination with sodium sulfite to form a redox system for the graft polymerization of the novel monomer 2-(furan-2-carbonyl) acrylonitrile onto chitosan under heterogeneous conditions. The grafted copolymers significantly led to a retardation in the bacterial and fungal growth and moreover, depicted good swelling properties indicating that they can also be employed as hydrogel materials.[32]

In 2010, Dipankar and co-workers employed potassium persulfate oxidation at optimized conditions for the depolymerisation of alginic acid biopolymer to prepare low molecular weight alginic acid based nanoparticles to serve as an effective drug delivery system for the management of drug resistant bacteria. The potassium persulfate concentration along with the pH and reaction time highly influences the alginic acid depolymerisation and hydrodynamic diameter decreases with increase in the concentration of potassium persulfate.[33]

Role of potassium persulfate in the degradation of organic contaminants and harmful chemicals

With the advancement, there is also an increase in the waste by product and pollutants in the environment. And thus increasing pollutants has surfaced a way for studying the use of potassium persulfate in improving treatment process against these hazardous chemicals. Moreover, recent waste water treatment processes rely on persulfate mediated advanced oxidation process (AOP) in order to degrade a wide range of organic pollutants and contaminants. An input of energy in the form of heat or photon leads to peroxide and activation in waste water treatment. Additionally, direct electrolysis or reduced metals, metal oxides are often adopted approaches that initiate peroxide bond breaking redox reaction. The basic principle behind these processes involves the production of $SO_4^{\bullet-}$ that initiates a cascade of reactions. This, in turn, leads to the formation of intermediate oxidants such as H_2O_2, $HSO_5^{\bullet-}$ and other reactive hydroxyl radicals, which could lead to the degradation of organic contaminants and harmful chemicals.[34]

Metformin, an anti-diabetic drug, is one of the pharmaceutical contaminant that can be found in local and clinic waste water, which are associated to influence the offset with biological systems and the human wellbeing. In 2018, Sawsen and Nadia used potassium persulfate as an electron acceptor in addition to hydrogen peroxide and sodium persulfate to improve the TiO_2 photo catalytic activity to investigate the degradation of metformin in aqueous medium under sunlight irradiation. Potassium persulfate was used as an additive in order to supplement the dissolved oxygen that would in turn lead to reaction rate improvement in the photo degradation by reacting with the electrons of the conduction band to form the intermediate radicals. In the process, the effect of different concentrations of potassium persulfate was investigated. The study showed that the degradation rate of metformin was increased at high potassium ion concentrations and achieved 91.6 % after 210 min of sunlight irradiation.[35]

In 2012, Subramanian and co-workers utilized potassium persulfate for degradation of Malachite green due to its genotoxic and carcinogenic properties. Malachite green was employed as a disinfectant, as a biocide in aquaculture, as an antihelminthic and also as a dye in paper and textile industries. Microbial assay revealed that the treatment of malachite green by potassium persulfate in presence of complex led to the removal of antibacterial activity indicating that this oxidation system has the ability to decrease the toxicity of malachite green towards the bacteria.[36]

In 2011, Saien and co-workers used potassium persulfate to facilitate the degradation of Triton X-100, most famous anionic surfactant that forms a significant fraction of dissolved organic pollutants in water ecosystems. It possesses wide practical applications in almost every type of liquid, paste and powdered cleaning compounds, ranging from heavy-duty industrial and agrochemical products to gentle detergents. 80.9 % degradation of TX-100 in 60 min and a complete degradation in only 30 min is achieved by applying homogenous AOPs, UV/H_2O_2 and UV/KPS under the optimum conditions of pH and temperature in addition to 270.3 mg/L of potassium persulfate.[37]

Summary and outlook

Potassium persulfate is widely used as a powerful oxidant in various organic transformations. Although metal persulfate seems to be a proprietary reagent for organic chemists, its application in areas other than organic chemistry has also been the subject of intensive investigation. This review highlighted the possible contributions of potassium persulfate in the field of biology and medical field by playing an important role for using as disinfection in biomedical field by degradation of contaminants and harmful chemicals. Further researches are needed in studying the utilization of metal persulfate that can contribute effectively in more spheres of environment and life as in the present scenario, The waste water treatment is on high upsurge, so we can use the potassium persulfate for the treatment of the water and in nano drug delivery system also.

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