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Zinc Oxide and Peroxide Nanoparticles - from Chemical Synthesis to Electrochemical Analysis and Wastewater Filtration

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Abstract

There is growing interest in the application of transition metal oxide and peroxide nanoparticles in materials science and industrial engineering. Transition metal oxide nanoparticles (TMONPs) can form materials with a wide range of unique chemical, electronic and physical properties for manufacturing various commercial products. Presently and for the foreseeable future, transition metal oxides are important constituents of energy production systems. They can be modified by various chemical, mineral, and polymeric substances, to produce nanocomposites that are suitable for the fabrication of more advanced materials. In this review article, discussion will focus on zinc oxide (ZnO) and zinc peroxide (ZnO_2) which are chemically related but technologically different in their applications. The toxicity of biochemically active ZnO nanoparticles has led to the development of new analytical methods with a focus on capillary electrophoresis with UV absorption or molecular fluorescence detection. The importance of wastewater analysis is emphasized with an outlook on the future perspectives in this fast-advancing research field. Next, the unique chemical properties and physical characteristics of ZnO_2 nanoparticles are introduced, followed by their latest applications in and modifications for scientific endeavors. Methods suitable for their incorporation in altering membrane properties to improve filtration performance are detailed. Major challenges and research endeavors are highlighted in the design of more effective membranes for wastewater filtration, hereby ameliorating the environmental toxicology of effluent contaminants. Polymeric membranes incorporated with composite zinc peroxide nanoparticles is proposed to be a good replacement of zinc oxide due to their strong oxidative properties involving a higher number of reactive oxygen atoms per molecule. The latest understanding of zinc oxide/peroxide nanomaterials toxicology is described, and best practices are crucial to control their environmental distribution. Last, the key research gaps that need to be addressed for future assessment of toxicological risks are unveiled.

Keywords: *Biosensors, Electrochemical analysis, Engineered composites, Membrane filtration, Nanoparticles, Toxicology, Transition metal oxides, Ultrafine dust, Wastewater, Zinc oxide, Zinc peroxide*

Introduction

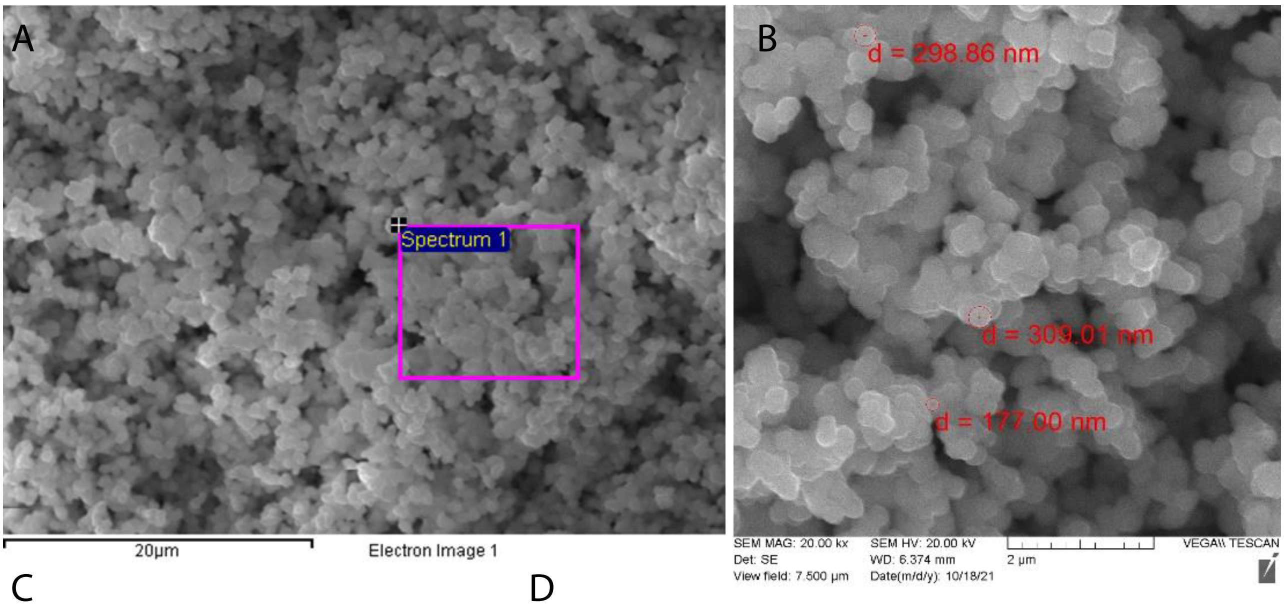
In recent years, advanced technology with nanoparticles have greatly sparked the interest of the scientific community. Their small sizes and tunable functional properties make them appealing as unique structures for biomedical applications, ranging from bioimaging, biosensing, drug delivery and theranostics. Material scientists have focused their research on the synthesis of transition metal oxide nanoparticles (TMONPs). Transition metal oxides exhibit unique physicochemical properties including catalytic function, ferroelectricity, piezoelectricity, magnetism, and supercapacitor performance. These oxides are fascinating to work with because their electrons interact strongly with each other, giving rise to a range of phenomenal effects such as high-temperature superconductivity and magnetoresistance. A comprehensive review summarizes different methods for synthesizing (TMONPs) as catalysts in oxidation reactions, and the unique role of metal oxide substrates in anchoring metal atoms for photocatalysis is emphasized. Biosynthesis of these nanoparticles by bacteria, fungi and plants can yield desirable crystallinity, diameters and morphologies if all process parameters (concentration, pH, temperature, time, and calcination temperature) are well controlled. Different TMONPs can be combined with other transition metal oxides to form nanocomposites that offer multiple synergistic advantages. They represent an important class of semiconductors finding applications in various major industries from solar energy transformation, magnetic storage media and electronic devices to photocatalysis. The development of novel electrochemical biosensors using morphologically varied transition metal oxides and their composites has highlighted the significance of TMONPs as promising electrode modifiers for the fabrication of electrochemical and biosensors. Reactivity of the metal oxide-water interface can be understood from the viewpoint of coordination chemistry, while the reactivity of a metal ion at a nanoparticle surface is compared to the reactivity of the same metal ion dissolved in an aqueous solution [1-12].

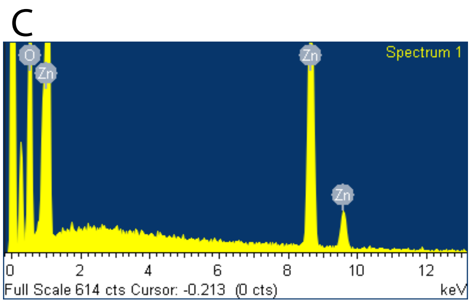
Nanotechnology is introducing many advantages over conventional methods of food processing, extending the shelf life, reducing deterioration, maintaining quality, and adding food values. Nanoparticles and nanomaterials improve barrier properties, detect pathogens, and alert the status of food. They will reduce the wastage of post-harvest loss of agriculture and horticulture produces. A substantial review has recently been published on the incorporation of transition metal oxides in the development of intelligent food nano-packaging. Peroxides are inorganic chemicals that contain a bivalent O-O group. These compounds release nascent oxygen readily and their major industrial applications include oxidizing agents, bleaching agents, and polymerization initiators. Chemical oxidation is one of the environmental site remediation methods that have emerged lately as a better alternative to traditional technologies. Nanosized oxidizing agents increase the ratio of surface to volume and hence the biodegradation speed for contaminants in soil and ground water. Sodium peroxide (Na_2O_2), sodium perborate, and sodium persulfate are common inorganic salts that react with water to produce hydrogen peroxide (H_2O_2). Other metal peroxides, such as BaO_2, CaO_2, CdO_2 and MgO_2, are highly stable and they promote the oxidation of organic substances only at higher temperatures. BaO_2, CaO_2, MgO_2, TiO_2 and ZnO_2 provide antibacterial applications in biomedicine. Tin peroxide (SnO_2) transforms to SnO when exposed to orange peel extracts with reducing ability. Zinc peroxide (ZnO_2) nanoparticles can be employed to prepare intelligent nano-packaging for better food preservation if they do not leach out from the packaging to the food [13-21].

Zinc Peroxide Nanoparticles

In 2021, zinc oxide and peroxide were the world's 776th most traded product, with a total trade of US$1.87B. The top exporters of ZnO_2 and ZnO were the Netherlands, Mexico, Canada, United States, and Peru. Both ZnO and ZnO_2 nanoparticles can be prepared from aqueous solutions containing zinc nitrate or formate using UV irradiation. When ZnO is treated with H_2O_2, an interfacial ZnO_2 layer forms to cover the nanoparticle surface. ZnO nanoparticles can be obtained by heat treatment of the peroxide above the transition temperature of 233°C, up to the decomposition temperature of 473°C. The weight loss due to the thermal decomposition of ZnO_2 into ZnO and O_2 at 250°C is considerably larger than the expected theoretical value of 16.4% just by oxygen release. ZnO_2 is a stronger oxidizing agent than ZnO. Decomposition of ZnO_2 to ZnO and O_2 resulted in the decrease of the band gap energy from 3.75 to 3.30 eV [22-28].

ZnO_2 nanoparticles were traditionally prepared by peptization of $Zn(OH)_2$ with the aid of H_2O_2 aqueous solution. They could be formed by a simple oxidation-hydrolysis-precipitation procedure, using zinc acetate as a precursor, hydrogen peroxide as an oxidizer, water as a preparation medium for hydrolysis, and polyethylene glycol as a stabilizer. ZnO_2 nanoparticles can facilely be synthesized from zinc acetate and H_2O_2 using a sol-gel method under ultrasound assistance [29-33]. Characterization by scanning electron microscopy and energy dispersive X-ray spectroscopy (Figure 1) shows an atomic ratio Zn/O of 2.01 and an average particle diameter of 304±5 nm. A





D

O K	32.94	66.75
Zn K	67.06	33.25
Totals	100.00	

Figure 1: (a, b) Scanning electron microscopy, and (c, d) energy dispersive X-ray spectroscopy of ZnO_2 nanoparticles facilely prepared in our lab by following Ramírez et al.'s sol-gel method under ultrasound assistance.

green method based on the reaction between $Zn_5(CO_3)_2(OH)_6$ powder and H_2O_2 in aqueous solution at room temperature can synthesize ZnO_2 nanoparticles. ZnO_2 nanoparticles can be prepared by the laser ablation of zinc in 30% H_2O_2 as another green technique using the fundamental wavelength (1064 nm) of a pulsed Nd: YAG laser, at a repetition rate of 15 Hz and laser fluence of 22 J/cm^2 for an ablation time of 10 min. Furthermore, the Leidenfrost dynamics occurring in an underwater overheated zone ensures eruption of nanoclusters towards a colder region, forming monodisperse nanoclusters of ZnO_2. Nowadays, ZnO_2 nanoparticles are commercially available, either bare or coated with an organic ligand shell of polyethylene glycol for stable dispersion in water, methanol, ethanol, acetone and dimethyl sulfoxide [34-38].

Unique Properties of Zinc Peroxide Nanoparticles

ZnO_2 is much more stable in aqueous solutions (as compared to calcium and magnesium peroxides) and it retains its peroxide content down to pH 6. At lower pH levels, H_2O_2 release is predictable as its dissolution product, Zn^{2+}, is highly soluble. Nanocrystalline ZnO_2 can be passivated against further oxidation by the addition of sub-stoichiometric amounts of potassium permanganate, which also increases the thermal stability of ZnO_2 [39,40].

ZnO_2 possess unique anti-bacterial, anti-corrosion, anti-fouling, and photocatalytic properties that are considered cost effective and environment friendly. They have been studied, due to their semiconducting and oxidizing properties, for various applications in optoelectronics, photocatalysis, sensors, biomedicine, and theranostics. Due to their large nonlinear optical susceptibilities, which are enhanced by two-photon electronic resonance, metal oxides are efficient sources of coherent anti-Stokes Raman Scattering (CARS). The FTIR spectrum of ZnO_2 nanoparticles shows a characteristic absorption peak at 435-445 cm^{-1}; the Raman spectrum shows characteristic peaks at 830-840 and 420-440 cm^{-1}. ZnO_2 nanoparticles exhibit photoluminescence with one strong emission band at 400 nm, one very weak emission band at 474 nm, and at 520 nm originating from the band edge and the oxygen vacancy. Polyvinyl alcohol/ZnO_2 nano-composite films have been engineered via casting. Their energy gaps decrease with increasing ZnO_2 concentrations to reach 2.80 eV at 2 wt.%, which are promising in anti-ultraviolet, opto-electronic, and optical limiting applications. A correlation exists between oxygen vacancies and the magnetization for pure ZnO_2 nanoparticles at room temperature. Coating of 15-20% ZnO_2 nanoparticles over graphene enhances magnetization more than 30 times due to the exchange interaction between localized electron spin moments resulting from oxygen vacancies at the surface [41-47].

ZnO_2 nanoparticles have reportedly oxidative stress mediated toxicity on various mammalian cell lines. Oxygen release from the biofunctionalized nanoparticles is tunable according to the solution pH. Antimicrobial tests at 37°C on bacterial species exhibiting different susceptibility to oxygen have confirmed the antimicrobial activity of ZnO_2 nanoparticles against *Enterococcus faecalis*, *Aggregatibacter actinomycetemcomitans*, *Porphyromonas gingivalis* and *Prevotella intermedia*. Accordingly, ZnO_2 showed effective antifungal activities, with a minimum inhibitory concentration (MIC) of 16 mg/L against *Candida* albicans. Histopathology assessment has confirmed the role of ZnO_2 nanoparticles in healing skin wounds. They exhibit angiogenic activity, due to onsite production of H_2O_2, for rapid tissue healing. ZnO_2 demonstrate antimicrobial, anti-elastase, anti-keratinase, and anti-inflammatory properties that are valuable for biomedical applications. They also inhibit bacterial biofilm formation and combat multi-drug resistant bacteria. A minimum concentration of ZnO_2 nanoparticles of 1 μg/mL inhibits the production of interleukin-1-β and interleukin 6 by peripheral blood mononuclear cells in the presence of lipopolysaccharides. ZnO_2 nanoparticles at a concentration of 2 μg/mL causes DNA damage in vitro; at a concentration of 5 μg/mL they promote protein aggregation and facilitate the production of protein complexes that may interfere with normal immune functions [48-54].

Applications of Zinc Peroxide Nanoparticles

Nanosized ZnO_2 is an efficient oxidant for the oxidation of aromatic alcohols to the corresponding carbonyl compounds selectively in excellent yields, using dimethyl carbonate as an environmentally benign solvent. ZnO_2 nanoparticles reactively adsorb chemical warfare agent surrogate of mustard gas, selectively oxidizing diethyl sulfide to diethyl sulfoxide and 2-chloroethyl ethyl sulfide to hydroxyethyl ethyl sulfide. Crosslinking of conventional/carboxylated nitrile rubber with ZnO_2 achieved total peroxide decomposition at vulcanization temperatures as low as 190-200°C [55-57].

Semiconducting CuO nanoparticles, as a CO_2 gas sensitive material, can with an organic binder and ZnO_2 for improved gas sensitive layer quality. The Lewis acid-base reaction between oxide oxygen and CO_2 has been proposed as sensing mechanism for the measurements in dry air, whereas the formation of surface barriers between nano-grains due to the reaction with CO_2 has been suggested for the CO_2 response under humid conditions [58].

ZnO_2 is a promising adsorbent nanomaterial for the removal of Congo red dye from contaminated water. The adsorption capacity is 208 mg g^{-1} within 10 min at pH 2-10. The adsorbent has a unique property to adjust pH within the 6.5-7.5 range irrespective of the acidic or basic nature of water. It is highly efficient even in the absence of sunlight to remove Congo red dye from contaminated water down to the permissible limits set by the World Health Organization and the United States Environmental Protection Agency. Crystal violet dye in industrial wastewater can be removed using sodium docusate-modified ZnO_2, attaining >99.5% adsorption efficiency in 5 min at pH ~10 as the zeta potential of ZnO_2 decreases from −15 mV at pH 3 to −60 mV at pH 9. The higher negative charge results in stronger electrostatic interaction with the dye. Synthetic graphite flakes can be treated with 3-mercaptopropionic acid, followed by functionalization with ZnO_2 nanoparticles, to efficiently remove As(III) and As(V). The adsorption data are best fitted with pseudo second order kinetic model and Freundlich adsorption isotherm, indicating chemisorption and multilayer adsorption on heterogeneous surface respectively. ZnO_2 nanoparticles, capped with polyvinyl-pyrrolidone to control the particle size, is an efficient material for the decontamination of cyanide from contaminated water by adsorption at pH 5.8-7.8 within 15 min [59-62].

As a catalyst for removal of reactive blue dye, a maximum degradation efficiency of 85% was achieved by ZnO_2 nanoparticles with polyethylene glycol, and 81% without PEG, after 120 min of photocatalytic reaction. ZnO_2 nanoparticles have excellent degradation efficiency of brilliant green dye, achieving 84-86% after 120 min of photocatalytic reaction at pH 6-7. Eco-friendly carbon quantum dots/ZnO_2 nanocomposite has been successfully synthesized for photocatalysis applications. It has higher efficiency than carbon quantum dots/TiO_2 for the removal of different dyes and high stability under UV-A light. Nitrobenzene photodegradation by ZnO_2 under UV lamps of 254 nm is optimal at pH 2, reaching up to 90% degradation in 2 h at 25°C [63-66].

For dental implants the accumulation of anaerobic bacteria is a main reason for peri-implant inflammation that can lead to implant loss. Decorating ZnO_2 by Glc-1P permits their uptake in the gram-negative oxygen-sensitive bacterial cells. ZnO_2 nanoparticles can be decorated with glucose 1-phosphate (Glc-1P) due to specific interaction of the phosphate function of Glc-1P with the nanoparticle surface. The anchored glucose molecules are accessible for specific interactions with lectin concanavalin A. Generation of ROS including hydrogen peroxide, hydroxyl radical, and peroxide anion can enhance the membrane permeability, cell wall damage, internalization of nanoparticles, and uptake of toxic dissolved Zn^{2+} ions. A ZnO_2-based theranostic nano-agent enhances oxidative damage to cancer cells by combining endogenous and exogenous reactive ROS. After uptake by cancer cells, the pH-responsive ZnO_2 nanoparticles, in addition to releasing exogenous H_2O_2, also provide Zn^{2+} to facilitate the production of endogenous $O_2^{\cdot-}$ and H_2O_2 from mitochondrial electron transport chain, enabling highly effective synergistic tumor therapy [67-69].

Zinc Oxide Nanoparticles

ZnO is a white powder that has two main lattice structures: hexagonal wurtzite and cubic zincblende. The hexagonal structure is commonly found, and both structures are insoluble in water with a solubility of 0.16 mg/100 mL at 30°C. The solubility of uncoated ZnO, as determined by the Zn^{2+} concentration in the aqueous solution, ranges between 20 and 47 mg/L. The solubility product constant K_{sp} is a useful parameter for calculating the aqueous solubility of sparingly soluble compounds. A comparison of dissolution rates shows that the ZnO nanoparticles have a higher dissolution rate than the bulk oxide. A new methodology for the *in-silico* assessment of the solubility of ZnO based on statistical thermodynamics, combined with density functional tight binding theory for the evaluation of the free energy exchange during the dissolution process. Complete ionic dissolution of ZnO is hindered by the formation of O^{2-} anions in solution, which are highly unstable. The dissolution rate will depend critically on the matrix with Zn ions and the mechanisms for diffusion or active transport of Zn^{2+} and O^{2-} ions in biological processes. Any mass fraction of Zn^{2+} ions removed or washed away will lead to further dissolution and eventually complete solubilization of the particulate fraction of ZnO. The fact that zinc-rich foods are mostly animal products suggests that vegetarians and vegans may have difficulty getting enough zinc in their diet. Zinc supplements are a great way to have the recommended levels of zinc in the body for stronger immune systems and improved muscle building. They are particularly useful for older adults (especially older men), who are more likely to have zinc deficiency [70-75].

Unique Properties of Zinc Oxide Nanoparticles

A variety of industries, including the automotive, concrete, cosmetic, pharmaceutical and textile, have used ZnO nanoparticles as a major material. The annual turnover of ZnO nanoparticles is over US$ 900,000/year, and the specific cost of their production is US$20/kg. Numerous synthetic techniques have been developed to meet the increasing demand for ZnO nanoparticles. These alternatives offer environmental and financial advantages associated with their commercial production. Biological synthesis uses plant extracts or microbes as green resources for the preparation of ZnO nanoparticles. Considerable investment to improve the performance of diverse nanocomposites allows rapid development of novel photocatalytic/photooxidizing degradation technologies for removing dyes in industrial wastewater [76-78].

Nowadays, ZnO nanoparticles continue to be a great attraction to researchers in various scientific endeavors based on their unique physicochemical properties. The natural bandgap (3.37 eV) and n-type conducting behavior of ZnO can be tuned by doping with metals/metal oxides and non-metals to replace Zn^{2+} and O^{2-} in the ZnO lattice, for various applications (such as solar cells, photocatalysis, medicines, light-emitting diodes, laser diodes, chemical and biosensors) owing to the direct influence of dopants on their electronic and physiochemical properties. A wide range of energy bandgaps (3-4 eV) is attained by green synthesis, indicating that ZnO nanoparticles can be employed in metal oxide semiconductor-based systems. The effectiveness of dye-sensitive solar cells is attributable to improved dye adsorption onto the nanoparticle surfaces. By adding ZnO in various amounts to a solution of polyvinylidene fluoride in 2-butanone during the fabrication process, followed by removing ZnO in an HCl bath once the organic solvent is evaporated, porous sensors can be made with different piezoelectric chains to control the piezoelectric coefficient. ZnO nanoparticles synthesized using the coprecipitation method present the best performance in catalysis, biosensing, imaging, drug delivery, and pollution absorption owing to their highest purity and crystalline phase, large Brunauer-Emmett-Teller surface area (~23 m^2g^{-1}) and pore volume in the mesoporous-macroporous structure [79-82].

ZnO nanoparticles have strong antimicrobial activity against a broad spectrum of bacteria (*P. aeruginosa*, *E. coli*, *A. baumannii*, *K. pneumoniae* and *Staphylococcus aureus*) and are effective against Hyalomma ticks. However, all fungal strains (*P. chrysogenum*, *A. niger*, *T. citrinoviride* and *A. fumigatus*) are resistant to ZnO nanoparticles. ZnO nanoparticles (1.5 mg/L) are protective against the detrimental effects of *Clostridium perfringes* type A infection in aquaculture. Their potential mechanisms of action against various kinds of viruses were discussed in a comprehensive review. The adoption of novel bio-assisted synthesis methodologies tailors the properties of ZnO nanoparticles to suit biomedical applications, underscoring their potential in cancer treatment towards MCF-7 breast cancer cell lines [83-87].

Applications of Zinc Oxide Nanoparticles

ZnO is produced synthetically for use as an additive in adhesives, antibacterials, baby powder, batteries, cement, ceramics, cigarette filters, cosmetics, ferrites, fire retardants, first-aid tapes, foods, glass, laser diodes, light emitting diodes, lubricants, ointments, paints, pigments, plastics, rubbers, sealants, semiconductors, solar cells, sun blocks, and wood products. Traditional uses of ZnO products include treating wounds following surgery and applying salves inside the mouth to treat ulcers or sores. Over 50% of ZnO is used in the rubber industry along with stearic acid for the vulcanization of rubber to produce tires, shoe soles, and even hockey pucks. ZnO nanoparticles have been added in food-packaging materials to stop food from spoiling. For use as binary/ternary composite anodes in lithium-ion batteries, ZnO has a higher theoretical capacity (978 mA.h/g) than many other transition metal oxides such as CoO (715 mA.h/g), NiO (718 mA.h/g) and CuO (674 mA.h/g). ZnO nanoparticles are extensively used in healthcare and environmental remediation applications attributable to their biodegradability. ZnO possesses unique biological properties for various antibacterial, antiinflammation, antitumor, and antiviral) applications. Addition of ZnO nanoparticles into crystal violet dye induces an alternative photoredox pathway, resulting in more generation of reactive oxygen species lethal to bacterial cells. This technique could be used to transform a wide range of bactericidal surfaces and contribute to maintaining low pathogen levels on hospital surfaces related to healthcare-associated infection. Hybrid ZnO-SiO_2 nanoparticles possess favorable characteristics for antifouling purposes. Self-cleaning and anti-fouling polymeric membranes for wastewater treatment are commercially fabricable with ZnO nanocomposites [88-100].

The formation and breaking of transition metal-carbon bonds plays a pivotal role in the catalytic oxidation of organic sulfides, alcohols, olefins, and alkanes. The textile industry is environment unfriendly due to the massive use of dyes and chemicals. Discharge of untreated textile wastewaters loaded with dyes not only contaminates the soil and water resources but also threatens the public health. ZnO nanorods can be used as a photocatalyst to degrade 65% of methylene blue in 50 min. Biochar-ZnO composites obtained by pyrolysis at 600°C can degrade 90% rhodamine B in 75 min, while ZnO can degrade only 38%. ZnO nanoparticles can be doped with Ni (3%), through combustion at 550°C, to improve the photocatalytic degradation of methylene blue and tetracycline [101-103].

Sodium (15%)-doped ZnO degrades 95% of methylene blue under visible light illumination in 180 min, with a rate constant of 1.7×10^{-2} min^{-1} and tenacious photostability. Green synthesis of ZnO nanoparticles is gaining huge attention via eco-friendly protocols that reduce the destructive effect of chemical synthesis. ZnO nanoparticles synthesized from Synadium grantii leaf extract with Cu dopant exhibit superior photocatalytic activity for indigo carmine, methylene blue and rhodamine B dyes. Gynostemma plant extract can be used in a co-precipitation method to synthesize ZnO nanoparticles for the photocatalytic decolorization of malachite green dye under UV illumination within 180 min. Biogenic ZnO nanoparticles can be synthesized by using *Pseudochrobactrum sp. C5* for catalytic degradation of dyes in wastewater treatment. Valorization of banana peel waste extract as the reducing and capping agents produces ZnO nanoparticles that show superior reusability and photodegradation efficiency for the removal of hazardous basic blue 9, crystal violet and cresol red dyes at pH 12 over irradiation time 90 min. Degradation of congo red by orange-peel-extract-biosynthesized ZnO nanoparticles via photocatalysis can remove 96% of the dye Photocatalytic degradation of rhodamine B dye in waste water and inhibition of butyrylcholinesterase, acetylcholinesterase and α-glycosidase enzymes are afforded by cauliflower-shaped ZnO nanoparticles synthesized using Alchemilla vulgaris leaves. Maximum photocatalytic degradation of pharmaceutical wastewater with ZnO was 40% and with TiO_2 is 33% at pH 9, following pseudo-first-order kinetics. Combined use of TiO_2/H_2O_2 is more effective than ZnO and TiO_2 alone, achieving 45% degradation. ZnO and TiO_2 can be used as catalysts for the degradation of dyeing factory effluents by the advanced oxidative process under UV irradiation at pH 3 for 8 h. Interestingly, a deposit of CdS nanoparticles on ZnO nanosheets provides excellent piezocatalytic efficiency for rhodamine B degradation under ultrasonic vibration. The nanocomposite of ZnO with porous hydroxyapatite (prepared from phosphate rock) improves the photodegradation of antibiotics in water and traps the by-products. An artificial neural network model can estimate the effect of different variables on AB113 dye removing decolorizing acid blue dye from textile wastewater in a sonophotocatalytic process. Reaction time, pH, ZnO dosage, ultrasonic power and persulfate dosage are optimized for maximum dye removal. After photocatalysis, if the treated water is discharged to the surface water along with the catalyst nanoparticles and degradation products, a resulting toxicity exists in the medium that can influence the lipid peroxidation and reduced glutathione in the aquatic vertebrates. Hence filtration is recommended before discharging, for separation of the catalyst nanoparticles. However, the filtration of nanoparticles from the treated water is costly and might outweigh the savings of energy [104-116].

Toxicology of ZnO Nanoparticles

Increasing production and application of transition metal oxide nanoparticles has raised concerns in regard to their environmental accumulation and toxicity in natural ecosystems. Nanoparticles are extensively studied for their chemical toxicology in aquatic microorganisms, agricultural products, fish, wildlife and humans. The uptake and accumulation of ZnO nanoparticles by aquatic organisms have considered the release of Zn^{2+} ions as well as the toxic mechanisms shared with other nanoparticles such as immunotoxicity, inflammation, lysosomal/mitochondrial damage, oxidative stress, programmed cell death, and redox activity. The growing usage of ZnO nanoparticles increases their release in municipal wastewater treatment plants. At 50 mg/L ZnO nanoparticles, both the granular activated sludge performance and the extracellular polymeric substances content are significantly reduced. This leads to decreases in the activities of ammonia monooxygenase and nitrate reductase. In addition, ZnO nanoparticles disrupt the cell membrane integrity and lead to bacterial cell death via intracellular ROS generation. After exposure to the nanoparticles, the bacterial community composition shifts to be dominated by Gram-positive bacteria. Antibacterial activity of ZnO nanoparticles is more pronounced with Gram-positive than Gram-negative bacteria. ZnO nanoparticles are biocompatible and effective as a food preservative against *Salmonella typhi*, *Klebsiella pneumoniae* and *Shigella flexneri*. They demonstrated significant antibacterial effects on various pathogenic bacteria in terms of zone-of-inhibition measured by the disc-diffusion method. When treated with ZnO nanoparticles (100-300 mg/L), significant reductions in marine microalgae *C. vulgaris* viable cells, LDH level, and non-enzymatic antioxidant glutathione are noticed while the activity of antioxidant enzyme superoxide dismutase and the level of lipid peroxidation significantly increase. ZnO nanoparticles possess antibacterial and antioxidant properties towards the remediation of hospital wastewater; the ones fabricated using Eriobotrya japonica leaves extract exhibit DPPH scavenging activity and are highly active against *S. aureus*, *P. multocida, E. coli* and *B. subtilis* strains. Redox imbalance, lignification and cell death cause reduction of root growth in wheat seedlings exposed to ZnO nanoparticles. Dietary exposure of carp to ZnO nanoparticles increases the aspartate aminotransferase activity significantly and decreases the alanine transferase activity significantly. ZnO nanoparticles act as a potent antidiabetic agent and severely elicit oxidative stress particularly at higher doses in diabetic rats (10 mg/kg). Initial exposure of human bronchial and pancreatic epithelial cells to oxidative stress sensitizes their subsequent response to cytotoxic challenge with ZnO nanoparticles. As *in vitro* model species human erythrocytes can be used to evaluate cytotoxicity, and human lymphocytes can be used for genotoxic studies. ZnO and TiO_2 nanoparticles result in 65% and 52% hemolysis at 250 ppm respectively, indicating cytotoxicity to human red blood cells. Both nanoparticles were found to generate ROS concomitant with depletion of glutathione and glutathione S-transferase levels. ZnO nanoparticles are significantly more genotoxic than TiO_2 nanoparticles at concentrations higher than 250 ppm. The nanoparticles preferentially kill cancerous cells over normal human cells. They enhance ultrasound-induced lipid peroxidation in the liposomal membrane. Two mechanisms underly the toxicity of ZnO nanoparticles: (i) generation of ROS and (ii) induction of apoptosis. The chemical toxicology of ZnO nanoparticles in adult male Wistar rats were investigated. All levels of zinc oxide nanoparticles had a significant impact on sperm quality and quantity. Significant toxicity effects of ZnO nanoparticles appeared at concentrations above 50 mg/kg body weight of animals. 200 mg/kg body weight resulted in increased total oxidant status and decreased total antioxidant capacity significantly. On the contrary, dietary supplementation of Nile tilapia with Se nanoparticles and ZnO nanoparticles induces synergistic effects that improve growth performance, blood health, and intestinal histomorphology. Seed priming with ZnO nanoparticles demonstrates beneficial effects of mitigating the phytotoxicity induced by Co stress in maize, significantly improving the plant growth, biomass, and photosynthetic machinery. Freshwater fish *O. mossambicus* fed with a supplemented diet of ZnO and Se nanoparticles raises the antioxidant response, boosts the immunity, and reduces the chance of getting infected by *A. Hydrophilia*. The entrance of ZnO or ZnS nanoparticles into freshwater systems may significantly impact the sedimentary microbial community structure and nitrogen cycling. Furthermore, they showed a strong anti-termite activity against *Heterotermes indicola* with a 100% mortality rate in 24 h [117-136].

Biosensors Incorporating Zinc Oxide Nanoparticles

Among all the optical biosensing systems, ZnO nanoparticles formed directly atop 3-aminopropyl triethoxysilane-treated Si substrates are more adhesive. Smaller particle sizes of ZnO will increase the fluorescence emission, eliminate several emission peaks, yield higher fluorescence quantum efficiency, and require lower excitation energy for fluorescence sensing. N-doped ZnO nanoparticles exhibit fluorescence emission at 385 nm (corresponding to the exciton absorption band) under excitation of 340 nm, responding with high selectivity and a detection limit of 4.9 μM for urea in blood serum. Self-assembly of diphenylalanine nanostructures in the presence of ZnO nanoparticles display distinctive luminescent emission at 550 nm that affords sensitive detection of trypsin down to 0.1 ng mL^{-1}. As a surface-enhanced Raman scattering substrate, ZnO tips can be decorated with gold nanoparticles to take advantage of the synergistic effect. Assay for nicotine demonstrates high sensitivity, reaching a lower detection limit of 8.9×10^{-12} mol/L and offering a linear dynamic range of 10^{-10}-10^{-6} mol/L. A localized plasmon-based fiber optic sensor can be immobilized with ZnO nanoparticles along with Au nanoparticles for the detection of p-cresol (a water pollutant) as low as 57 μM. A field effect transistor device consisting of ZnO nanoparticles and glutathione-S-transferase in the composite channel can successfully detect and quantifies glutathione in solution and in cancerous cells. The glucose content in food samples can be determined using ZnO nanoparticles, with a correlation coefficient of 0.9812 at 3.5 mM-27.8 mM concentrations [137-143].

A novel electrochemical sensor made by drop casting zinc oxide nanoparticles and electropolymerizing glutamic acid can detect sodium dodecyl sulfate with excellent selectivity via molecular imprinting. ZnO was overlaid on the interdigitated electrode of an electrochemical DNA biosensor to detect sequence complementation from *Ganoderma boninense*. ZnO nanoparticles prove to be excellent for doping carbon dots in electrochemical biosensor applications. Chemical vapor deposition of ZnO nanoparticles on an aluminum foil working electrode successfully sensed cysteine electrochemically. Smartphones can be combined with screen-printed electrodes or interdigital electrodes for in-situ electrochemical detection. The electrodes are often modified with biomaterials, chemical materials, and nanomaterials (such as ZnO) for biosensing to monitor ascorbic acid, dopamine, glucose, levodopa, and uric acid in point-of-care testing. Aluminum doping can be attained by radio frequency magnetron sputtering of ZnO nanoparticles deposited on a glass substrate for biosensor applications. Four different H_2O_2 biosensors have been designed using ZnO nanoparticles, multiwalled carbon nanotubes, Prussian blue, ionic liquid and horseradish peroxidase. The best analytical performance offers a linear dynamic range of 9.99×10^{-8}–7.55×10^{-4} M, detection limit of 1.37×10^{-8} M, and sensitivity of 17.00 μA mM^{-1}. A laser scribed graphene-$ZnFe_2O_4$ electrochemical aptasensor for acute myocardial infarction screening has been developed for detecting the cardiac troponin-I biomarker, with a limit of detection of 0.001 ng/mL and a sensitivity of 19.3 μA/(ng/mL). The Ag-ZnO-graphene oxide/glassy carbon electrode exhibits high sensitivity, detection limit of 0.02 μM, and fast response within 3 s owing to the efficient oxidation of diclofenac sodium at 0.25

V. Trimetallic Ni/Ag/Zn oxide composite-modified glass carbon electrode has good sensor sensitivity of 0.96 μA/μM cm^2 and detection limit of 0.3 μM for dopamine. ZnO-reduced graphene oxide-Au nanoparticles can modify a screen-printed electrode for fast electrochemical detection of dopamine in biological samples. A uric acid biosensor was constructed with nafion/uricase/ZnO nanorods-ZnO nanoparticles on a fluorine-doped tin oxide electrode. Differential pulse voltammetry demonstrated linearity over a wide concentration range (0.01-1.5 mM) with a high sensitivity (345 μA mM^{-1}cm^{-2}) and low limit of detection (2.5 μM). A glassy carbon electrode modified with carbon nanotubes, cytochrome C and ZnO nanoparticles has good sensitivity for the detection of streptomycin in pharmaceutical samples. The highly sensitive interface of penicillinase@CHIT/PtNP-ZnO/ZnHCF/FTO electrode shows a linear response and good limit of detection (0.1 μM) in antibiotics in forensic samples. Biosensors based on ZnO and NiO nanostructures decorated with Au nanoparticles have opened the doors to detect volatile organic compounds using electrochemical methods. Biomass carbon derived from cassava and its composites with ZnO nanoparticles can be synthesized for biosensing due to their low cost and resource availability [144-159]. In our lab, screen-printed electrodes are modified by a deposit of ZnO nanoparticles from aqueous suspension onto the graphite working electrode surface. After drying, a sample solution containing sodium metabisulfite analyte in 1 M KCl can be placed on top for chronoamperometry using the Homianze μEA 160C electrochemical analyzer. A typical current-time curve is obtained as shown in Figure 2a, which is ready for data analysis in accordance with the Cottrell equation as shown in Figure 2b.

Self-cleaning and Anti-fouling Polymeric Membranes for Wastewater Treatment and Analytical Separations

A recent trend in nanotechnology shows the application of nano-based materials, such as nano-adsorbents, nano-metals, nano-membranes, and photocatalysts, in water treatment processes. Nanomaterials typically have high reactivity and a high degree of functionalization, large specific surface area, and size-dependent

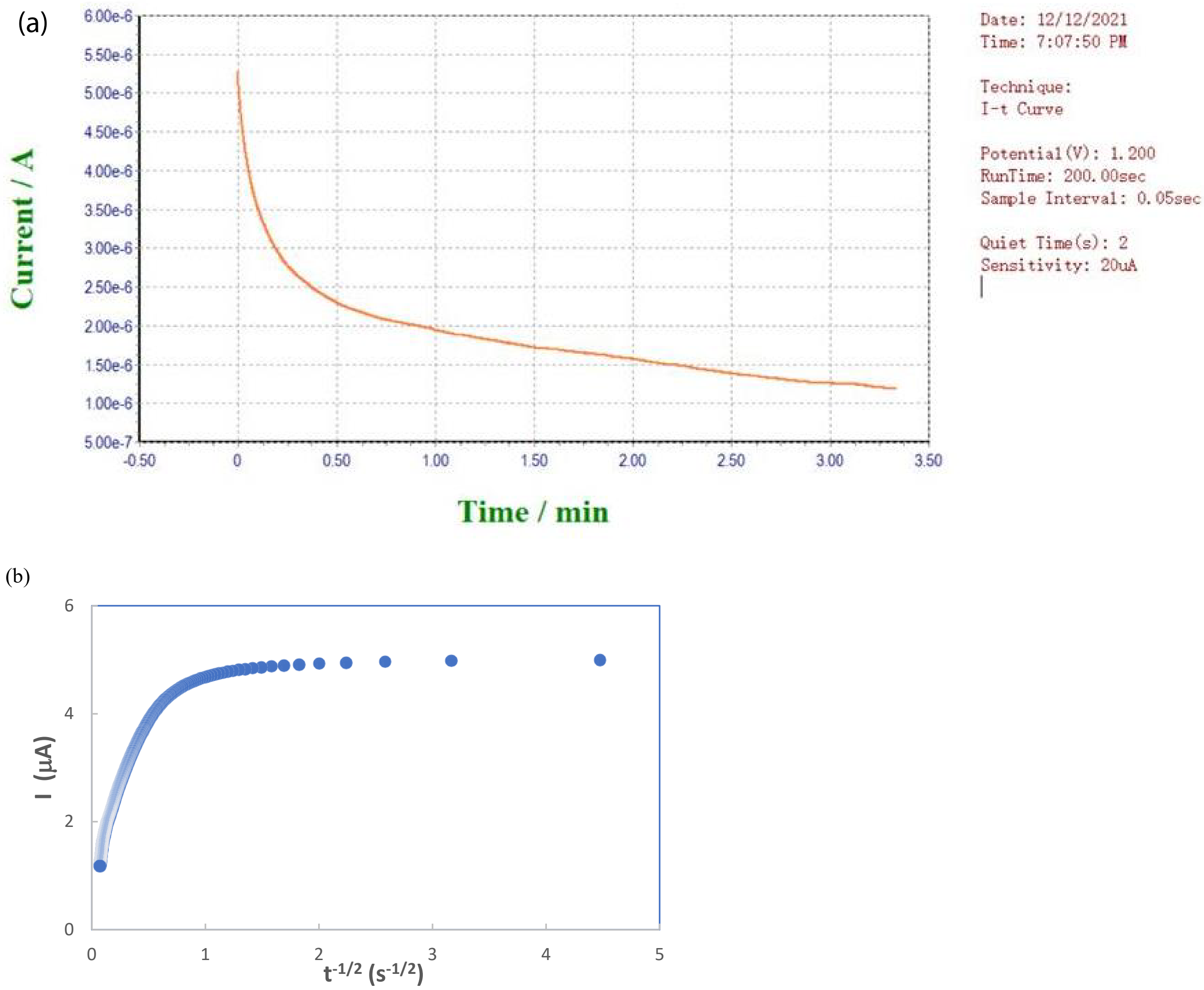


Figure 2: (a) Screen shot of current-time curve obtained in our lab from sodium metabisulfite with ZnO nanoparticles deposited on graphite electrode. (b) Plot of current vs. inverse square root of time for Cottrell analysis.

properties which makes them suitable for applications in wastewater treatment and for water purification. Nanostructured catalytic membranes, nanosorbents and nanophotocatalyst-based approaches to remove pollutants from wastewater are eco-friendly and efficient, but they require more energy and more investment in order to purify the wastewater. Current and potential applications of nanoparticles and nanotechnologies in wastewater treatment as well as challenges have been reviewed on the basis of bibliometric results [160-165]. Self-cleaning surfaces have attracted significant attention in both the scientific and industrial communities [166]. In the past decade, transition metal oxide nanoparticles have extensively been incorporated with polymeric membranes for water treatment. Special emphasis is given here to their anti-fouling and self-cleaning properties when used also in the preparation of wastewater samples before chemical composition analysis. Various forms of copper, titanium dioxide and zinc peroxide were tested against microbial fouling and microbiologically influenced corrosion. Their incorporation into polyethylene (high density) and fiber-reinforced plastic provides surface protection. Wastewater treatment is currently a crucial topic worldwide due to global human population growth (83 million annually), industrial downstream contamination, and weathering degradation of polymers [167-170]. Various water treatment techniques are being advanced due to the rising concern of drinking water scarcity and safety. Besides conventional water treatments, the pressure-driven water purification technology has attracted attention due to its efficiency and received substantial applications. Pressure-driven membranes can be classified into microfiltration, ultrafiltration, nanofiltration, and reverse-osmosis. These membranes are used to separate ions, macromolecules, suspended particles and nanomaterials from water. Organic polymeric membranes are extensively used for commercial purposes due to their excellent physical, chemical, and mechanical characteristics. However, membrane fouling occurs due to their hydrophobic nature plus bacterial accumulation and is limiting their sustained operation over time. Regarding membrane fouling, a combination of polymer and nanoparticles is suggested to be a practical strategy for enhancing membrane hydrophilicity. Incorporating nanoparticles into polymeric membranes is becoming a trend in membrane technology. Polymers and metal oxides are becoming popular membrane filtration materials for wastewater treatment due to their surface functionality, large surface area, and unique optical/paramagnetic properties. Under visible light conditions, the polymer-metal oxide nanocomposite membrane affords superior photodegradation activity toward organic pollutants. Transition metal oxides have been evaluated by many researchers during the last decade for wastewater reclamation, as self-cleaning and anti-fouling agents, to utilize their surface mobility, magnetic and optical properties. Recently, a review on polymer nanocomposite membranes based on metal oxide nanoparticles was published in the field of ultrafiltration membrane technology [171-194].

ZnO nanoparticles have been extensively used by scientists and researchers, known to be inorganic, hydrophilic, low-cost, and green (environment-friendly) material. Fluoride contamination of water is a serious problem in the world, and zinc oxide nanoparticles are the best adsorbent for the removal of fluoride from water and wastewater, with an adsorption capacity of 100 mg/g. In wastewater treatment processes, ZnO nanoparticles exert a negative impact on the sludge flocculation performance but do not significantly impact the sludge sedimentation behavior. A decrease of the tyrosine protein-like substance level is probably the key reason for the decreased ζ potential in the loosely bound extracellular polymeric substances, which eventually induces a decline of the sludge flocculation performance under the ZnO stress. A novel deflocculant ZnO/chitosan nanocomposite film in disperser pretreatment enhances the energy efficiency of anaerobic digestion by achieving 99% solubilization of organics. In addition to the anti-fouling performance, ZnO nanoparticles also provide photocatalytic self-cleaning ability to the polymeric membranes. Hence, ZnO-incorporated composite membranes were considered an emerging topic in membrane technology. Modification of polyvinyl chloride membrane using ZnO nanoparticles is very effective for municipal wastewater treatment in the presence of ferric chloride coagulant. The nanocomposite membrane did not adsorb the sludge inside the pores, hence substantially limiting the membrane fouling. Polyvinylidene fluoride membrane with high hydrophilicity was reported to be developed through the conglomeration of ZnO and graphene oxide. Anti-fouling properties, porosity, water flux and wettability were improved to attain a stable effluent quality (0.6 NTU). Combination of graphene oxide and ZnO nanoparticles on polysulfone membrane surface improves the membrane performances to treat petroleum refinery wastewater in terms of higher porosity, increased hydrophilicity, better mechanical strength, reduced water contact angle, increased water uptake ability, higher permeate flux, rejection of total dissolved solids, and improved antifouling properties. Unfortunately, no sustainable membrane systems are yet fully established due to their huge energy requirements for partial removal or degradation of trace organic compounds. Impregnation of ZnO-graphene also reduces polyethersulfone membrane-solute and membrane-foulant hydrophobic interactions. ZnO incorporation enhances the hydrophilicity and improves the anti-fouling property of polyether sulfone membranes. A mean pore size of 0.64 nm and good humic acid rejection make the hybrid membrane well suited for nanofiltration in wastewater treatment and water reclamation. Multifunctional nanofibrous membranes with sunlight-driven self-cleaning performance for complex oily wastewater remediation can be constructed with an Ag/ZnO layer on the porous polyacrylonitrile nanofiber substrate. The membranes demonstrate excellent mechanical strength, superhydrophilic (water contact angle = 0°), underwater superoleophobic (contact angle = 154°) properties, high permeation flux (>619 $Lm^{-2}h^{-1}$) and separation efficiency (>99.7%) for various oil-in-water emulsions [195-204].

The effect of photoactive semiconductor catalyst (TiO_2 and ZnO) on the anti-fouling and self-cleaning properties of polyether sulfone composite membranes (14% by weight) was studied with different concentrations of graphene oxide. The hydrophilicity of composite membranes improved as compared to neat membrane; however, graphene oxide-TiO_2 functionalized membranes showed the lowest flux. Incorporation of CuO nanoparticles in polymeric membranes for water treatment is a potential solution for biofouling formation. Promising results have been reported for antibacterial/

antifouling effects, increased hydrophilicity, water flux improvement, contaminant rejection capacity, structural membrane parameters, and reduction of concentration polarization. TiO_2 nanoparticles have been added to improve the self-cleaning and anti-fouling ability of ultrafiltration polymer membranes through their photocatalytic activity. Immobilization of TiO_2 nanoparticles on membrane surfaces was investigated to reduce organic fouling effects in a bioreactor by increasing the membrane hydrophilicity. *Cajanus cajan* seed extract and carbon nanoparticles reformed the hydrophobic PVDF membrane to hydrophilic. Introduction of TiO_2 (0.02% by weight) into the membrane rendered it bi-functional, thus achieving 85% rejection of Cr(VI) and 92% reduction to Cr(III) in tannery wastewater. Ag_2O, Fe_2O_3 and ZrO_2 nanoparticles can be incorporated to improve the performance of polymeric filtration membranes due to their effects on permeability, selectivity, hydrophilicity, conductivity, mechanical strength, thermal stability, antiviral, and antibacterial properties. However, they might cause membrane deterioration. Thus, careful selection is required to choose the best composition of metal oxide nanoparticles for individual polymeric membranes. The advantages and disadvantages of Ag_2O, CuO, Fe_2O_3, TiO_2, ZnO and ZrO_2-incorporated polymeric membranes for water purification have been compared in a new review. Their characteristics (antibacterial property, anti-viral property, conductivity, contaminants rejection, flux permeation, hydrophilicity, mechanical strength, permeability, surface charge, and thermal stability as shown in Table 1) can help decide on the best modification towards achieving sustainable and cost-effective treatment operations. Bimetallic transition metal oxide nanoparticles have attracted many researchers due to their salient features and characteristics over mono metallic oxide nanoparticles. PES ultrafiltration membranes were fabricated using the phase inversion technique (most commonly used technique to fabricate polymeric porous membranes with a large form of structure) with a composite of Fe_2O_3-Mn_2O_3 nanoparticles as modifier. Those membranes showed an excellent porosity (74%), high water flux (398 L/m^2h), and better antifouling ability. Protein-based filtration tests showed an improved flux recovery ratio in protein separation and water treatment applications [205-214].

Fabrication of transition metal oxide nanoparticles-modified polymeric membranes to make them operation-sustainable cost-efficient is challenging. Transition metal oxide nanoparticles have many interesting functional properties. However, integrating nanoparticles into a membrane remains a challenge. Atomic layer deposition and sequential infiltration synthesis were explored for the modification of polymeric membranes and fabrication of novel mesoporous structures. Fouling is a major problem that hinders the operation of membrane filtration processes. Bio-fouling causes performance degradation and elevates energy consumption due to blockage of membrane pores. In addition, it increases the frequency of membrane cleaning and reduces the membrane life span, thereby leading to higher maintenance and operation costs. Antibacterial membranes are considered an attractive strategy to retard biofouling. ZnO is reported to be useful as an anti-fouling agent in polymeric nanofiltration and reverse-osmosis membranes. Instead of ZnO, ZnO_2 nanoparticles can be incorporated to make nanofiltration and reverse-osmosis polymeric membranes for better retardation of fouling since ZnO_2 is a stronger oxidizing agent than ZnO and can produce free radicals and other reactive oxygen species to inhibit growth of microorganisms. The operating temperature of nanofiltration and reverse-osmosis is typically within 25-65°C, which is far below the transition temperature (233°C) of ZnO_2. Hence, ZnO_2 nanoparticles embedded in the polymeric membrane are completely stable during wastewater treatment. ZnO_2 has photocatalytic self-cleaning property that would make it a strong modifier over ZnO in the fabrication of polymeric membranes. Importantly, these membranes can greatly facilitate the preparation of samples for instrumental analysis of emerging contaminants by removing microplastics (plastic particles smaller than 5 mm) that exist in wastewater and marine environments, including pharmaceuticals, personal care products, perfluoroalkyl substances, organophosphate flame retardants, illicit drugs, and isoprostanes in wastewater as biomarkers of oxidative stress during COVID-19 pandemic. A complete summary of recent advances and latest studies in the fabrication, modification, and industrial application of ZnO photocatalysts is available for further reading. Black TiO_2 nanotube array can be employed as both photocatalyst and electrocatalyst to degrade dissolved organic matter in coking wastewater [215-227].

Table 1: Transition metal oxide nanoparticles-incorporated polymeric membranes.

Transition Metal Oxides	Polymeric Membrane	Advantages	Limitations
ZnO	Polysulfone, polyurethane, polyvinylidene difluoride	Antibacterial, anti-corrosion, anti-fouling, environment-friendly, hydrophilic, low-cost, mechanical strength, self-cleaning (photocatalytic activity).	Not stable (photocatalytic property), mildly toxic.
TiO_2		Antibacterial, anti-corrosion, anti-fouling, hydrophilic self-cleaning (photocatalytic activity).	High doses may induce cytotoxicity, not stable (photocatalytic property).
Fe_2O_3		Abundantly available, can remove heavy metals, magnetic properties, mechanical strength, non-toxic.	Nanoparticles tend to agglomerate easily.
CuO		Antibacterial, anti-corrosion, anti-fouling, compound rejection capacity, hydrophilic, improving water flux, mechanical strength.	Low-quality nanoparticles are produced via physical synthesis, toxic chemicals are used if produced through chemical synthesis.
Ag_2O	Pressure retarded osmosis membranes	Almost non-toxic, anti-fouling, antimicrobial, resistant to corrosion, stable.	Membranes are sensitive to nanoparticle concentration.
ZrO_2	Novel membranes	Capable of treating saline water, high-temperature stability, high water retention capacity.	Fouling susceptibility, expensive raw materials.
Fe_2O_3–Mn_2O_3	Polyether sulfone	Antifouling, minimal irreversible fouling, excellent water flux, improved recovery for protein separation.	Agglomeration of nanoparticles.
TiO_2-ZnO composite membrane		Increased hydrophilicity, anti-fouling, self-cleaning properties, photocatalytic activity.	Low flux.

Analytical Methods for Transition Metal Oxide Nanoparticles

New developments have recently been reported concerning the chemical analysis of transition metal oxide nanoparticles in environmental water due to their biochemical toxicity. A unifying methodology for the selective detection of transition metal oxide nanoparticles in water, as well as sensitive determination of environmentally toxic and biochemically active contaminants that are bound on them, is urgently needed. In our lab, new analytical methods have undergone intensive development in the last ten years with a focus on capillary electrophoresis with UV and molecular fluorescence detection (Figure 3). The toxic effects of these emerging contaminants have already been verified by Health Canada and Environment Canada using bioassays. The methodology under intensive development in our research lab begins with a sample treatment step that encapsulates all waterborne nanoparticles/nanomaterials into lecithin liposomes. Centrifugation concentrates the loaded liposomes, and the supernatant water is withdrawn for instrumental analysis by liquid chromatography with detection by tandem mass spectrometry. Next a surfactant disintegrates the liposomes and isolates lecithin from the nanoparticles. Contaminants are desorbed from the nanoparticle surfaces using coordination chemistry, biochemical interaction, laser photo/photothermal chemistry, aerosol nebulization, and electrospray ionization. The desorbed contaminants can be analyzed, either immediately or after separation by capillary electrophoretic/gel filtration/liquid chromatography, by spectrofluorometric and mass spectrometric detection. Optical incoherent scattering and electrochemical chemistry can be adapted to detect the nanoparticles and desorbed contaminants at trace to ultratrace levels [228-238].

Transition metal oxide nanoparticles are increasingly used as a solid carrier in the formulation of numerous drug products. They end up in waste streams, consequently infiltrating the aquatic environments and drinking water resources. Detection of nanoparticles in wastewater requires more advanced analytical methods than conventional water analysis, to prevent the ecosystem of plants and animals from unintended exposure to the released pharmaceuticals. Determination of nanoparticles in drinking water has important implications for protecting the public health sustainability. Unfortunately, many emerging contaminants are not yet stipulated in water quality regulations due to a lack of monitoring technology. Hence, there is an urgent need to develop new analytical methods that can monitor emerging contaminants in water resources. The interplay of nanoparticles, environmental pollution, and health risks is key to all industrial, environmental, and drinking water treatment regulations. A unifying analytical methodology will help scientists and engineers strengthen their control of nanoparticles in freshwater sources for drinking water treatment plants. New endeavors must challenge the traditional notion that environmental toxicological events involve only dissolved contaminants. Rather, environmental toxicology can involve a complex assortment of nanoparticles and associated contaminants whose combined effects on biological and mammalian cells are continuously evolving. The precise toxico-pathogenic effects of ZnO nanoparticles on the cardiovascular system under normal and cardiovascular disease risk factor milieu include down regulation of vascular development and elevation of oxidative stress in the heart tissue. Both endothelial nitric oxide generation and cardiac Ca^{2+}-ATPase activity are significantly suppressed; the cardiac mitochondrial swelling is enhanced [239].

Wastewater Analysis

The nanoparticles released from different nanomaterials used in our household and industrial commodities find their way through waste disposal routes into the wastewater treatment facilities and end up in wastewater sludge. Further escape of these nanoparticles into the effluent will contaminate the aquatic and soil environment. Polyacrylic acid nanomembranes can be used as nano-filters to isolate and remove Ag and TiO_2 nanoparticles in aqueous environmental samples using pressure-driven flow, with a filtration efficiency of >99%. The phytoremediation potential of *Myriophyllum spicatum* L. for removal of ZnO nanoparticles in tap water ranges between 29% and 70%, and slightly higher in pond water. Wastewater treatment plants are a primary source of many contaminants to the environment.

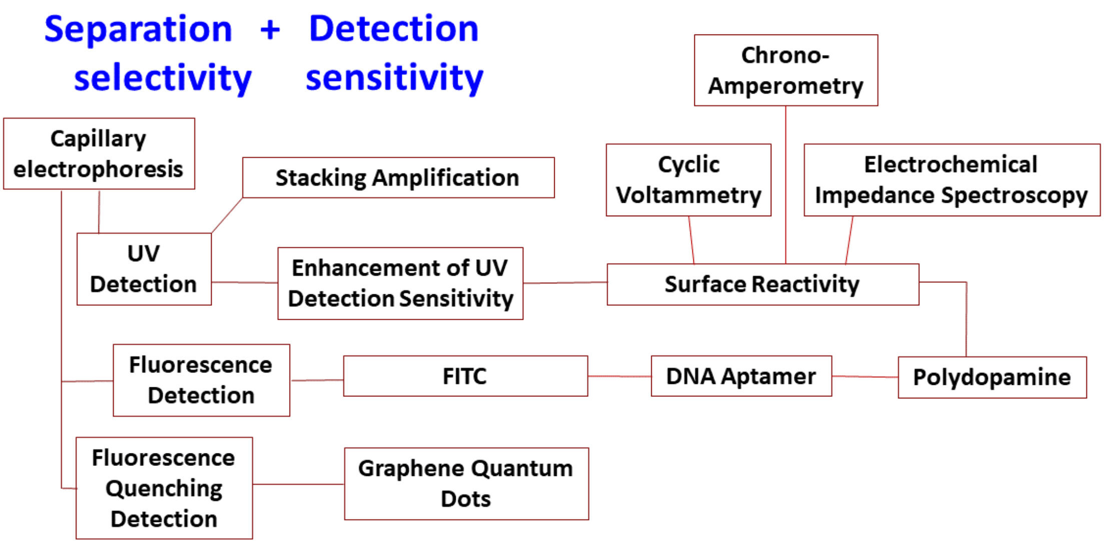


Figure 3: Development of new analytical methods for transition metal oxide nanoparticles in our lab.

Processing complex mixtures of waste, they can result in the continuous discharge of bioactive and endogenous compounds into sensitive aquatic ecosystems. Wastewater analysis has been demonstrated to be a cumulative approach for assessing the overall patterns of alcohol, drugs, tobacco and xenobiotic use by a population at the community level. Hospital wastewater, for one, is regarded as a very important source of fluoroquinolone antibiotics (ciprofloxacin, norfloxacin, and ofloxacin) in the aquatic environment. The development of analytical methods is crucial for the detection of oxidative stress biomarkers in wastewater, using ultra-high-performance liquid chromatography coupled with tandem mass spectrometry and solid phase extraction. Mixed liquor can be collected from the secondary aeration tank while effluent wastewater is collected after the secondary settling tank in a wastewater treatment plant. Mixed liquor is the wastewater which leaves the aeration tank after biological treatment before going into the secondary settling tank for the suspended solids to sediment, while effluent wastewater is the ultimate treated wastewater which is discharged to the river from the treatment plant. Obviously, mixed liquor has much higher levels of suspended solids and relatively higher dissolved carbon content compared to effluent wastewater. Nanoparticles from thirteen different elements were determined, throughout the full-scale wastewater treatment process, by using single particle inductively coupled plasma mass spectrometry. Samples of the influent, post-primary treatment, effluent of the activated sludge process, as well as reclaimed water were analyzed. The incidence of metal-based nanoparticles decreases significantly after the conventional wastewater treatment train, and they are smaller in the effluent (<180 nm) than in the influent (<300 nm). However, anaerobic digesters store high nanoparticle concentrations. Hence, the disposal of sludge needs to take this into account to evaluate the risk of nanoparticles release to the environment [240-248].

The potential use of 8-iso-PGF2α as a sewage biomarker for assessing the status of community health was investigated by liquid chromatography-high resolution mass spectrometry coupled to immunoaffinity clean-up and β-glucuronidase treatment. Urinary excretion provides a mechanism for the entry of isoprostanes to wastewater treatment plants and subsequently the wider environment, where they may initiate a cycle of oxidative stress in aquatic biota. Additional isoprostanes may be produced within these organisms, further perpetuating this cycle of toxicity. An analytical method for their detection in wastewater, based on solid phase extraction and gas chromatography mass spectroscopy, involves a deconjugation treatment with -glucuronidase to increase the concentration of isoprostanes available for detection. The low ng/L range of concentrations of human metabolic biomarkers and the complex matrix composition pose bioanalytical challenges related to sample preparation, detection and quantification. A sensitive liquid chromatography-mass spectrometry method for the detection and analysis of opioid biomarkers has been validated according to the European Medicines Agency guidelines; Oasis HLB cartridges are useful for sample concentration. Ion pairing liquid chromatography with alkanesulfonates coupled to tandem mass spectrometry is valid for the analysis of aminoglycosides (veterinary antibiotics) in wastewater samples after addition of the ion paring salt directly into the raw or treated wastewater samples. Surface-enhanced Raman spectroscopy is good for the detection of methamphetamine based upon the assembly of Au@Ag core-shell nanoparticles on a disposable glassy nanofibrous electrospun paper matrix that gives strong scattering signals. Microplastics are generated while polishing eyeglass lenses and a huge amount of nanoplastics (<1 μm) passes through the conventional wastewater treatment process in considerable amounts. Microplastics (with adsorbed heavy metals) can be quantified in the wastewater by mass balance measurements using membrane filtration with polyaluminum chloride coagulation. The transport of nanoparticles in various wastewater treatment processes is fully discussed in another review [249-255].

Air Pollution Remediation and Quality Monitoring

One of the most favorable environmental applications of nanotechnology has been in air pollution remediation in which different nanomaterials are used. Nanoparticles have initiated the advancement in new and low-cost techniques for environmental pollution control including air pollution. Metal oxide nanofibers have demonstrated to be effective for air pollution remediation in the form of filter, catalyst, catalyst support, and photocatalyst. Fibrous metal oxide has several advantages including surface area, mechanic strength, chemical stability, thermal stability, and photocatalytic ability. In the field of selective reduction of nitrogen oxide, a catalyst with low cost, low toxicity, high activity, and good selectivity for N_2 is needed to replace the high-cost and high-toxicity vanadium catalyst. Low-cost spinels MFe_2O_4 (M = Cu, Mn, and Zn) can be synthesized for this application, and $MnFe_2O_4$ exhibits the best activity (99.9%) and selectivity (95.7%) at 100°C [256-258].

Nanocomposites have distinctive physical and chemical properties that result in their use in the construction industry as innovative materials. Addition of nanoparticles can bring many important properties to the bulk construction and insulation materials. Unfortunately, release of ultrafine dust to the air environment has harmful impacts on human health. Nanoparticles can enter the human body through the skin, inhalation, and ingestion. Exposure to nanoparticles can cause serious respiratory, cardiovascular, skin, and nerve related diseases. It can pass through various mammalian membranes, or be absorbed in them, to cause various inflammatory reactions and fibrosis. Pneumoconiosis refers to a class of interstitial lung diseases caused by the inhalation of airborne dust and fibers. Engineered nanoparticles, owing to their high reactivity, can initiate inflammatory responses that trigger metastasis. Human exposure to nanoparticles can cause various health implications such as DNA damage and cell death. A global regulatory policy needs to be framed to assess the toxicity, risk and approval of nanoparticles used in the construction industry. The current OSHA standard for ZnO fume is 5 mg/m^3 of air averaged over an eight-hour work shift. NIOSH recommends that the permissible exposure limit be changed to 5 mg/m^3 averaged over a work shift of up to 10 hours per day, 40 hours per week, with a short-term exposure limit of 10 mg/m^3 averaged over a 15-minute period. It would be scientifically interesting to investigate the percutaneous absorption of transition metal oxide nanoparticles following exposure to road dust powder [259-264].

Semiconducting metal oxide gas sensors have been developed for environmental gases including CO_2, O_2, O_3 and NH_3; highly toxic gases including CO, H_2S and NO_2; combustible gases such as CH_4, H_2, and liquefied petroleum gas; and volatile organic compound gases. Nanomaterial enabled sensors are applied for the detection of harmful gases such as H_2S, SO_2, and NO_2. An ultrafast sensor has been developed for trace-level detection of NH_3 gas using ZnO nanoparticles, with ultra-fast response (5 sec) and recovery time (8 sec) at 5 ppm. Dopants can enhance the performance of semiconductor metal oxides for gas sensing applications by changing their microstructure morphology, activation energy, electronic structure, and band gap of the metal oxides. In some cases, dopants create defects in semiconductor metal oxides by generating oxygen vacancy or by forming solid solutions. To date, very little is known about the magnitude, patterns, and associated risks of human exposure to microplastics, particularly in the indoor environment. This is a significant research gap given that people spend most of their time indoors, which is exacerbated over the past year by COVID-19 lockdown measures [265-269].

Conclusion

This scientific field possesses immense potential that may provide incredible technological advances soon. The research findings covered in this review article could open many doors to new endeavors. Having more reactive oxygen atoms per molecule, ZnO_2 can be considered as a stronger oxidizing agent than ZnO. This unique property makes its nanoparticles an excellent candidate for potential breakthroughs in analytical, biomolecular, food, material, and separation sciences. In addition, mono-dispersed ZnO_2 nanoparticles could be coupled with a magnetic core to produce nanocarriers for in-situ disruption of cancer cells. High-performance nano-filtration and reverse-osmosis could be developed with ZnO_2 for fouling remediation with self-cleaning feature. For future applications, intelligent antibacterial food nano-packaging could undergo new developments through incorporation of ZnO_2 nanoparticles to the packaging film. It will become more and more important that the presence of ZnO/ZnO_2 in wastewater is detected and quantitated for the protection of environmental sustainability and public health. The knowledge gap in this dynamic field, as highlighted in this review, will require novel research work.

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Competing Interest Statement

The authors have no competing interests to declare.

Data Availability Statement

The raw/processed data required to reproduce our findings in Figure 2a/2b cannot be shared at this time as the data also forms part of an ongoing study.

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