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ENVIRONMENT-FRIENDLY SYNTHESIS OF TITANIUM OXIDE (TIO_2) NANOPARTICLES, CHARACTERIZATION, AND ITS APPLICATIONS

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ABSTRACT

Green processes are the best method to prepare nanoparticles that are economical and environment-friendly. Current reviews focus on the synthesis of TiO_2 NPs and their potential applications. Plant extracts (leaves, fruit, shell and flower) and other biological sources are used in the environment-friendly synthesis of TiO_2 NPs. Through a reduction mechanism, TiO_2 NPs are made by reacting titanium salt with these biological sources. These biological sources are rich of metabolites. The TiO_2 NPs have exclusive morphology and its surface chemistry shows a wide range of applications. The TiO_2 NPs are mainly spherical with a particle size range between 1-100 nm, there is always a rich source of metabolites. They have been studied as a photocatalyst to remove toxic element from water. TiO_2 NPs are also used for photocatalytic removal of dye RG-19 and many other dyes which are toxic and hazardous pollutants. TiO_2 NPs exhibit potent antiparasitic and antibacterial activities against parasitic insects and pathogenic microorganisms. These TiO_2 NPs exhibited size and dose-dependent antimicrobial and antiparasitic activities.

KEY WORDS : Environment-friendly synthesis, Titanium oxide nanoparticles, Plant part extracts, Photocatalysis, Antimicrobial activity, Antiparasitic activity

INTRODUCTION

Nanotechnology has a lot of potential for scientific discovery in the twenty-first century. The nanoparticles are 1 nm to 100 nm in size. The metal nanoparticles (MNPs) are made using a variety of techniques. Chemical reduction, sol-gel technique, sonochemical, electrolysis, arc discharge, environment-friendly and microorganisms are just a few of them. Researchers are particularly interested in the environmentally friendly production of nanoparticles utilising plant extracts (Manjare and Chaudhari, 2020a; Manjare and Chaudhari, 2020b; Manjare *et al*., 2020). Environmentally-friendly nanoparticle synthesis is a simple and cost-effective method that has been employed in numerous environmental evaluation and treatment processes (Pushpamalini *et al*., 2020).

Nadeem *et al.* (2018) have taken a review on the synthesis of TiO_2 NPs using plant part extracts. The highlight of the study was the synthesis of TiO_2 NPs and its applications in antimicrobial and photocatalytic activity. The review was unable to clarify the mechanism by which TiO_2 NPs are prepared and how they are used for different bioactivities. The photocatalytic activity of TiO_2 NPshas been reported by various authors for reduction of various dyes and compounds (Nadeem *et al.*, 2018). The main aim of this review to analyse the betterment of environment-friendly methods and its application in photocatalytic and antimicrobial analysis.

Goutam *et al.* (2018) published research paper an environment-friendly method for producing TiO_2 NPs. These TiO_2 NPs are utilised to remove chromium and chemical oxygen demand from tannery effluent that has been secondary treated (Goutam *et al*., 2018). The results against antibacterial activity are determined using TiO_2 NPs. Gram-negative bacteria were used to test the antibacterial activity (*E. coli and P. aeruginosa*). For the gram studied above, these have remarkable results (Ajmal *et al*., 2019).

In this review, we discussed the environment-friendly synthesis of TiO_2 NPs using various plant part extracts and their applications in the photocatalytic, antibacterial and antiparasitic activities.

REVIEW

Synthesis of Titanium oxide (TiO_2) nanoparticles

Synthesis of metal nanoparticles (MNPs) using green chemistry is the utilization of a set of principles that might eliminates the use or generation of hazardous substances in the design. Chemical products may have synthesized by using green chemistry principles. This method has been proposed as cost-effective and environment-friendly alternative to chemical and physical methods. The employment of green synthesis of nanoparticles is bound to the use of biological hosts including bacteria, algae, fungi, yeast and plant part extracts. Plant extracts containing phytochemical components as reducing and capping agents cause the reduction of metal ions and stabilization of nanoparticles using different mechanisms including steric stabilization, electrostatic stabilizationand stabilization by hydration forces and Van der Waals forces. The well stabilized nanoparticles play an important role in their functions and applications as catalyst in organic synthesis (Kaur *et al*., 2021).

Irshad *et al*. (2021) published a review on the synthesis, characterisation and advanced applications of TiO_2 NPs. Fourier transform infrared spectroscopy (FTIR), UV-Visible, X-ray diffraction (XRD), High resolution scanning electron microscopy (HR-SEM), Energy dispersive X-ray spectroscopy (EDX) and Transmission electron microscopy (TEM) have all been used to characterise TiO_2 NPs (Irshad *et al.,* 2021). Environment-friendly synthesis is the best pathway for the synthesis of TiO_2 NPs. In this review, we are focussing environment-friendly synthesis of TiO_2 NPs using plant part extract.

Environment-friendly synthesis of TiO_2 NPs using plant part extracts

Nadeem *et al.* (2018) conducted a review on the environment-friendly production of TiO_2 NPs. The production of TiO_2 NPs from various biological sources was one of the highlights of this review. The photocatalytic, antiparasitic and antibacterial activities of TiO_2 NPs seems likely to produce noteworthy outcomes. Many authors in this review were unable to describe the mechanism of TiO_2 NP production. The synthesis of TiO_2 NPs is attributed to different metabolites found in plant component extracts. Figure 1 depicts the mechanism of TiO_2 NP production utilising various plant extracts.

Goutam *et al.,* (2018) used *Jatropha curcas* leaf extract to make TiO_2 NPs. UV-Visible, FTIR, XRD, SEM and Brunauer-Emmett-Teller analyses were used to characterise the biosynthesized TiO_2 NPs. Environment-friendly produced TiO_2 NPs had an average crystalline size of 13 nm, a surface area of 27.038 m^2/g, a pore size diameter of 19.100 nm and a total pore volume of 0.1291cm^3/g, respectively. The green synthesis of TiO_2 NPs using a biodegradable and non-toxic extract from *J. curcas* leaves is a very promising method. Figure 2 depicts a possibleme chanism for the production of TiO_2 NPs in the presence of hydroxyl groups from *J. curcas* L. extract as a capping agent.

Ajmal *et al.,* (2019) synthesized TiO_2 NPs using methanolic extract of *P. domestica* L. (Plum), *A. deliciosa* (Kiwi) and *P. Persia* L. (Peach) fruit peels. The biosynthesized TiO_2NPs were characterized by XRD, FTIR and SEM. The size of TiO_2 NPs synthesized from plum, kiwi and peach were found to be 47.01, 54.17 and 85.1 nm respectively. The SEM images of all three TiO_2 NPs revealed cylindrical in shape. XRD spectrum of identified TiO_2 NPs found

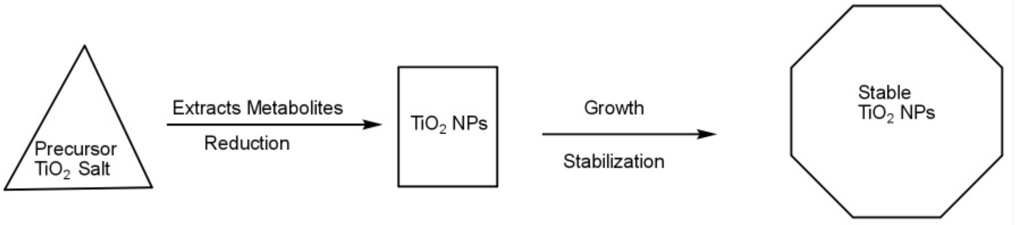

Fig. 1. Reduction mechanism of TiO_2 NPs (Nadeem *et al.,* 2018)

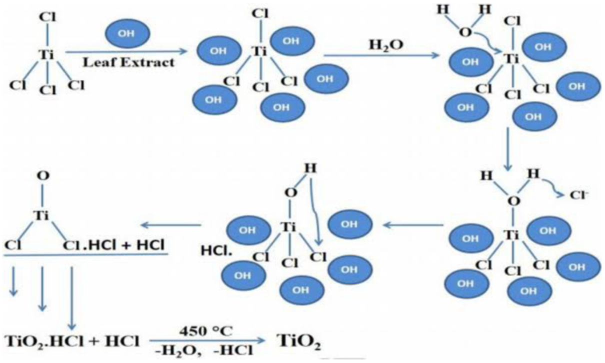

Fig. 2. Reaction mechanism for the formation of TiO_2 NPs in presence of hydroxyl group of leaf extract of *Jatropha curcas* (Goutam *et al.*, 2018)

noncrystalline in nature. All the TiO_2 NPs exhibited the size and dose dependent antibacterial and antioxidant activities.

Kaur *et al.* (2019) used titanium (IV)-isopropoxide (TTIP) to make TiO_2 NPs from *Carica papaya* leaf extract. The cages like morphology with spherical nanoparticles of particle size 15.9 nm was observed during characterisation. TiO_2 NPs have a mesoporous structure, and at a dosage of 25 mg, they have a fantastic removal effectiveness for RO-4 dye (91.19%). The TiO_2 NPs were obtained by using *Carica papaya* extract. These TiO_2 NPs were calcined at 400 °C. The obtained TiO_2NPs were crystalline in the form.

Bhullar *et al.* (2021) published a paper describing a fast green production of TiO_2 NPs (Bhullar *et al.*, 2021). The extracts of black pepper, coriander and clove were used to make TiO_2 NPs in this study. For the production of TiO_2 NPs, they use the sol-gel process. XRD, HR-TEM and SEM analyses were used to characterise the nanoparticles. The produced nanoparticles have sizes ranging from 6.7 to 8.3 nm as determined by HR-TEM. This shows that the XRD analysis and the HR-TEM results are in good agreement. Nanomedicine and other drug delivery applications benefit greatly from these nanoparticles. Figure 3 depicts the general concept for making titanium oxide nanoparticles.

Green synthesis of TiO_2 NPs using *Moringa oleifera* leaf extract has been reported by Patidar and Jain (Patidar and Jain, 2017). Structural properties of the synthesized nanoparticleswere characterized by using XRD analysis. XRD analysis showed that the anatase TiO_2NPs sample having trigonal structure. The energy band gap of TiO_2 NPs was calculated 3.9 eV which was greater than bulk TiO_2 having band gap of 3.2 eV. In 2019, Kulkarni *et al.* synthesized TiO_2 NPs using *Azadirachta indica* (Neem) leaf extract and characterization was carried out using XRD, Zetasizer, SEM and AFM (Kulkarni *et al.*, 2019). The TiO_2NPs were prepared by 24 hours heating of *Azadirachta indica* (Neem) extract and TTIP mixture. The average size of TiO_2NPs measured by Zetasizer was 56.13 nm which was spherical in shape and having a fine particle.

Subhapriya and Gomathipriya, (2018) synthesized TiO_2 NPs by *Trigonella foenum-graecum* leaf extract. The 15 ml of leaf extract was added to 0.5 M solution of titanium-oxy-sulphate and stirred for 15 minutes for synthesis of TiO_2 NPs and these nanoparticles were characterized using FTIR, UV, XRD, SEM, EDX and HR-TEM. Synthesized TiO_2NPs nanoparticles showed significant antimicrobial activity. Likewise, *Psidium guajava* leaf extract was used to synthesis TiO_2NPs which showed spherical shape and clusters with an average size of 32 to 58 nm which was reported by Santoshkumar *et al.* (2014).

The synthesis of TiO_2 NPs using plant part extracts is simple and environment-friendly. Therefore, there are more reports on the production of TiO_2 NPs using plant part extracts. Some of them with mechanism of bioreduction are summarized in Table 1.

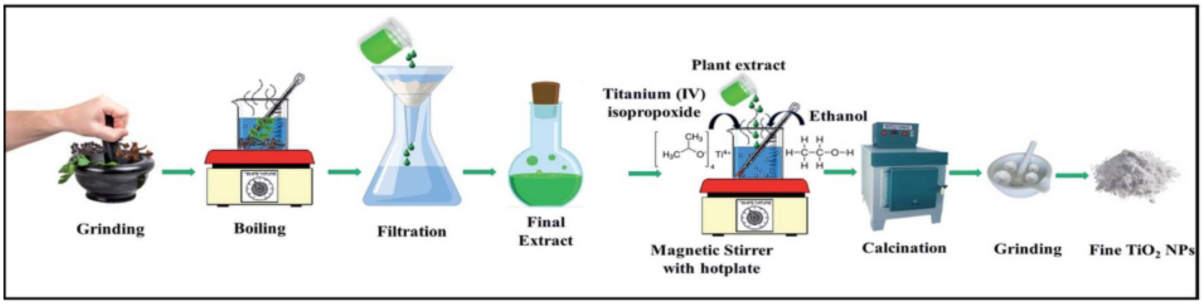

Fig. 3. Environment-friendly and cost effective route for the green synthesis of nanoparticles using plants part extracts (Bhullar *et al*., 2021)

Applications of biosynthesized TiO_2 NPs

The biosynthesized nanoparticles are of great interest as these exhibit exclusive morphologies and surface chemistry. These biosynthesized TiO_2NPs have a wide range of applications in antiparasitic, antimicrobial and antioxidant activity. The most important application which is widely exploited is their incredible photocatalytic activity to clean the contaminated water and in the degradation of various dyes and pollutants. The TiO_2 NPs have been reported by various authors for reduction of various dyes and compounds (Jadoun *et al.*, 2021).

Photocatalytic activity of TiO_2 NPs

Many industries discharge their waste into the river. Hazardous contaminants, such as toxic dyes and hazardous organic and inorganic chemicals, are found in these effluents. These effluents may be highly stable, posing numerous dangers to aquatic life.

An environment-friendly approach for the synthesis of TiO_2 NPs using *Jatropha curcas* leaf extract was reported by Goutam *et al.*, 2017 (Goutam *et al.*, 2018). The synthesized TiO_2 NPs were used as a solar photocatalytic removal of Cr^{+6} from tannery wastewater. According to TWW wastewater, the chemical oxygen demand removal effectiveness of TiO_2 NPs is 82.26%, while Cr removal efficiency is 74.48%. The removal of Cr from TWW wastewater employing TiO_2 NPs as a photocatalyst is observed to be very slow when compared to chemical oxygen demand. The following is the mechanism for the conversion of Cr^{+6} to Cr^{+3}:

$$TiO_2 + hv \rightarrow h^+ + e^-$$

$$Cr_2O_7^{2-} + 14h^+ + 6e^- \rightarrow 2Cr^{3+} + 7H_2O$$

$$2H_2O + 4h^+ \rightarrow O^2 + 4H^+$$

$$H_2O + h^+ \rightarrow OH^- + H^+$$

$$OH^- + \text{organics} \rightarrow CO_2 + H_2O$$

$$H^+ + \text{organics} \rightarrow CO_2 + H_2O$$

In an acidic solution, these TiO_2 NPs demonstrate good photocatalytic activity against a hazardous reactive green-19 dye (Kaur *et al.*, 2019). When compared to artificially generated TiO_2 NPs, these nanoparticles remove 98.88% of the hazardous reactive green-19 dye. 30 mg of catalyst and 120 minutes of UV light irradiation were employed to

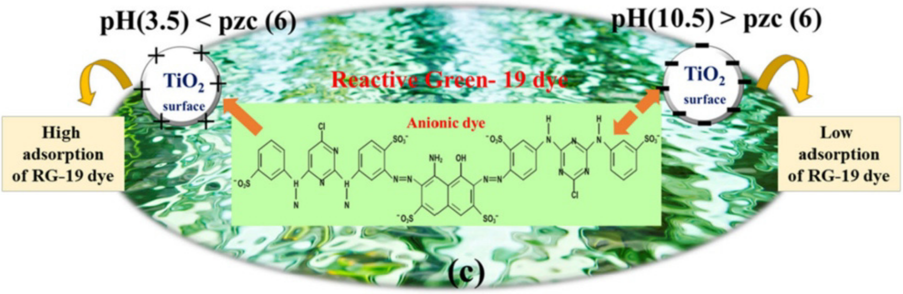

Fig. 4. Effect of pH on adsorption of dye on TiO_2 NPs (Kaur *et al.*, 2019)

Table 1. TiO_2 NPs synthesized using plant part extracts

Plant Part Extract	Metal salt	Size in nm	Morphology	Reference
Jatropha curcas leaf	$TiCl_4$	13	Spherical	Goutam *et al.*(2018)
L. Siceraria leaf	TTIP	8.6	Spherical	Kaur *et al.* (2021)
Carica papaya leaf	TTIP	15.6	Spherical	Kaur *et al.* (2019)
Trigonella foenum leaf	$TiOSO_4$	42-66	Spherical	Subhapriya and Gomathipriya, (2018)
P. guajava leaf	$TiO(OH)_2$	32.58	Spherical	Santhoshkumar *et al.* (2014)
Moringa oleifera leaf	TTIP	12.22	Tetragonal	Sivaranjani and Philominathan (2016)
Azadirachta indica leaf	TTIP	56.13	Spherical	Thakur *et al.* (2019)
S. cumini leaf	TTIP	18	Spherical	Sethy *et al.* (2020)
N. oleander leaf	TTIP	65-75	Spherical	Vijitha *et al.* (2019)
Jasmine flower	TTIP	32-48	Spherical	Aravind *et al.* (2021)
Cucurbita pepo seeds	$TiCl_3$	-	Tetragonal	Abisharani *et al.* (2019)
Glycosmis cochinchinensis leaf	$TiO(OH)_2$	45	Spherical	Rosi and Kalyanasundaram, (2018)
E. H. Jaub root	$TiO(OH)_2$	25.2	Spherical	Nasrollahzadeh and Sajadi, (2015)
Arbor-tristis leaf	TTIP	75	Spherical	Sundrarajan and Gowri, (2011)
Caricaceae (Papaya) shell	TTIP	15	Spherical	Saka *et al.* 2022

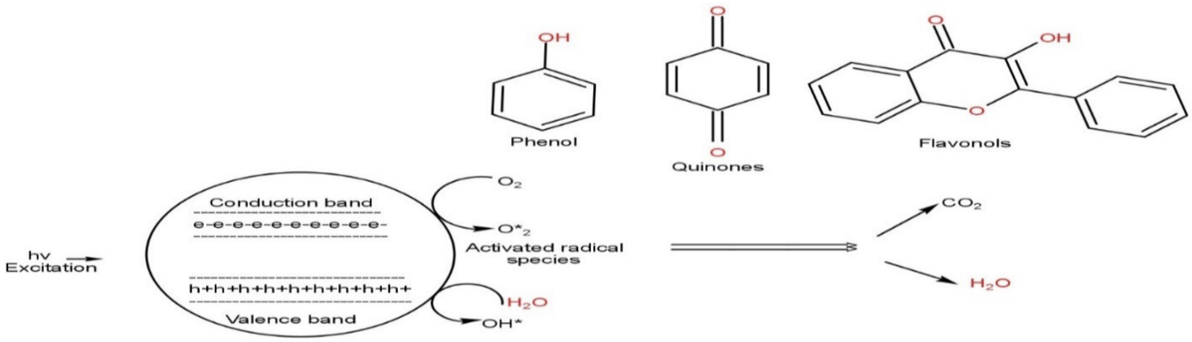

Fig. 5. Photocatalytic mechanism for pollution removal by using TiO_2 NPs under light (Kaur *et al*., 2019)

remove reactive green-19 dye. In acidic medium, UV absorption is higher for harmful RG-19 dye than in basic medium, resulting in dye photodegradation. The effect of pH on dye adsorption on TiO_2 NPs is shown in Figure 4.

Kaur *et al*. (2019) reported an environmentally friendly TiO_2 NPs production utilising *Carica papaya* leaf extract. The photodegradation of an aqueous solution of RO-4 dye with synthesized nanoparticles showed improved outcomes at various pH values of the dye solution. The percentage dye elimination by nanoparticles increased from 85.18% to 92.16% when the dosage of TiO_2 NPs was raised from 15 mg to 30 mg. The dye removed is 91.19% at the optimum dosage of TiO_2 NPs, which is 25 mg. Figure 5 depicts the photocatalytic pathway for pollution removal utilising TiO_2 NPs under light.

Sethy *et al*. (2020) published research paper an environmentally friendly synthesis of TiO_2 NPs using *Syzygium cumini* extract for photocatalytic removal of lead in explosive industrial waste water in the same year. The TiO_2 NPs were spherical and aggregated into an irregular structure with an average diameter of 18 nm. These TiO_2 NPs eliminate lead from explosives and industrial waste water in a cost-effective manner. TiO_2 NPs remove 82.53% of lead from explosive industrial effluent and 75.5% of chemical oxygen demand. Various parameters of water before and after treatment are summarized in the Table 2.

Table 2. Various parameters of water before and after treatment of TiO_2 NPs (Sethy *et al.*, 2020)

Physico-chemical parameter	Before treatment	After treatment
pH	7.6	7.8
Lead (ppm)	8.621	1.5
COD (mg/L)	8450	2004
Colour (a.u.)	Yellow (0.15)	Light yellow (0.07)

Aravind *et al.* (2021) reported a biosynthesis of TiO_2 NPs using Jasmine flower extract. They examined the photodegradation of methylene blue dye with the help of UV-Visible irradiation technique using biosynthesized nanoparticles. Which results in the degradation of methylene blue to leuco methylene blue. Biosynthesized TiO_2NPs showed maximum degradation efficiency of 89% under 120 minutes of irradiation.

Antimicrobial and antiparasitic activity

When compared to chemically manufactured TiO_2 NPs, green synthesised TiO_2 NPs demonstrated superior antimicrobial or antibacterial action against negative microorganisms. *S. saprophyticus*, *B. subtilis*, *E. coli* and *P. aeruginosa* were used to test the

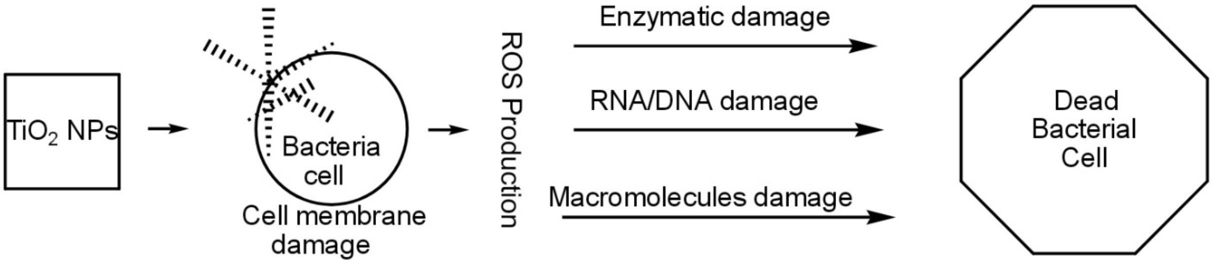

Fig. 6. Mechanistic representation of green synthesized titanium nanoparticles effect on the bacterial cell (Rosi and Kalyanasundaram, 2018)

Table 3. ZOI against different bacteria with TiO_2 NPs concentration (Ajmal *et al*., 2019)

Bacteria	Type gram	Zone of inhibition	NPs conc. in ìg/ml
E. coli	Negative	3-20 mm	12.5-100
P. aeruginosa	Negative	2-19 mm	12.5-100
A. Substilis	Positive	17-19 mm	100

antibacterial activity of green produced TiO_2 NPs against a variety of harmful bacteria.

Ajmal *et al.* (2019) synthesized TiO_2 NPs using fruit peel agro waste. The zone of inhibition against different bacteria is shown in Table 3. It found that TiO_2 NPs exhibited good antimicrobial activity against all gram negative bacteria with which *E. coli* being most affected.

The biogenesis of TiO_2 NPs by *Trigonella foenum-graecum* leaf extract was reported by Subhapriya and Gomathipriya (2018). The antibacterial activity of these nanoparticles against *Y. enterocolitica* (10.6 mm), *S. faecalis* (11.6 mm), *S. aureus* (11.2 mm), *E. faecalis* (11.4 mm) and *E. coli* (10.8 mm) is noteworthy. The inhibition zone for TF-TiO_2 NPs is observed between 8.5 mm-11.5 mm. Because of the hydroxyl group, TiO_2 NPs have the ability to disintegrate the outer membrane of bacteria, resulting in the microorganisms death.

An environment-friendly fabrication of TiO_2 NPs using *Psidium guajava* leaf extract was reported by Santoshkumar *et al.* (2014). The biosynthesized TiO_2 NPs (20 µg/mg) observed maximum zone of inhibition against *S. aureus* (25mm) and *E. coli* (23 mm). The disk diffusion method was performed against the pathogenic strains of bacteria.

Aravind *et al.*, (2021) designed an environmentally friendly synthesis of TiO_2 NPs that exhibit good antibacterial activity. The inhibitory zone for gram negative bacteria like *E. coli* and *Klebsiella* was 14 mm and 12 mm respectively, when these nanoparticles were used. These TiO_2 NPs have a remarkable zone of inhibition for gram positive bacteria like *S. aureus*, which is 8 mm. Reactive oxygen species including hydroxyl group and superoxide were used to breakdown bacterial cell walls which results in death of microbes.

Rosi and Kalyanasundaram, (2018) developed an environment-friendly synthesis of TiO_2 NPs using *G. cochinchinensis* leaf extract. These TiO_2 NPs have antibacterial action against both gram positive (*S. saprophyticus* and *B. Subtilis*) and gram negative bacteria (*E. coli* and *P. aeruginosa*). The comparison reveals that gram negative bacteria have the largest inhibitory zone. Figure 6. depicts a plausible mechanistic pathway for TiO_2 NPs impacts on bacterial cell walls.

Benelli *et al.* (2017) found that metal nanoparticles were efficient against a variety of parasitic larval and adult insect species. Green TiO_2 NPs have also been discovered to be potent larvicidal agents against a variety of parasitic bug species. Reduced levels of biochemical parameters related in growth and development, such as proteins, lipids, lactate dehydrogenase, alkaline phosphatase and acid phosphatase, are among the subcellular events that lead to mortality as shown in Figure 7. TiO_2 NPs were found to be effective against a variety of parasites including *B. ovis*, *Aedes aegypti*, *H. maculate* and others. This is more effective against Zika virus.

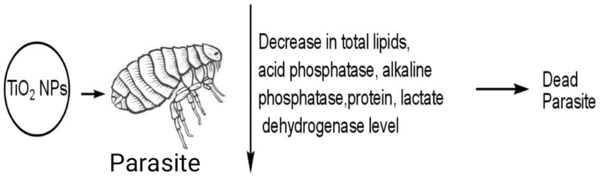

Fig. 7. Effect of biogenic TiO_2 NPs on larvas biochemical factors (Benelli *et al*., 2017)

These findings show that green produced TiO_2 NPs have exceptional photocatalytic activity, however the reducing phenomena is yet unknown and needs to be investigated further. It possesses a wide range of antibacterial and antiparasitic properties. It's also employed as an antioxidant and in the creation of gel-based ointments(Jadoun *et al.*, 2021).

CONCLUSION

The focus of this review study was on the environment-friendly production of TiO_2 NPs using a variety of plant extracts and their applications. The formation of TiO_2 NPs was initially verified using morphological and spectroscopic measurements, followed by a change in the reaction mixtures colour. Characterization of biosynthesized TiO_2 NPs was done using XRD, UV-Visible, FTIR, SEM and TEM. This review emphasises TiO_2 NPs photocatalytic, antibacterial and antiparasitic properties. In order to eliminate hazardous metals from TWW, TiO_2 NPs were used as a photocatalyst. It's also utilised as a photocatalyst to remove poisonous dyes and other harmful contaminants. Future expectations from environment-friendly nanoparticle synthesis are finding new routes of synthesis and the applications of these nanoparticles in catalysis and the biomedical area will rise tremendously.

Abbreviations

1) TiO_2NPs Titanium oxide nanoparticles
2) FTIR-Fourier transform infrared spectroscopy
3) TEM-Transmission electron microscopy
4) XRD-X-ray diffraction spectroscopy
5) SEM-Scanning electron microscopy
6) EDX-Energy dispersive X-ray spectroscopy
7) TTIP-Titanium (IV)-iso-propoxide
8) RG-19-Reactive green – 19
9) COD-Chemical oxygen demand
10) TWW-Tannery wastewater
11) ZOI-Zone of inhibition
12) ROS-Reactive oxygen species

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