NAAS: 6.85

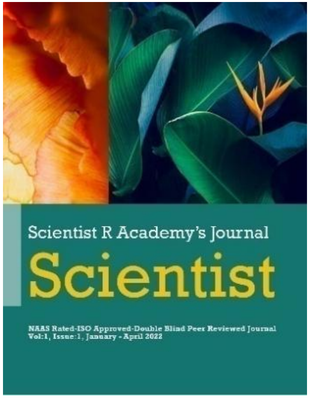
(QZ CNR 2022273, 0890-3670)

Volume 1, Issue 2, May-August 2022

Scientist

The official Journal of Scientist R Academy

DOI: https://doi.org/10.5281/zenodo.6924075



Research Article

BIOSYNTHESIS OF TITANIUM DIOXIDE NANOPARTICLES AND THEIR APPLICATION AS ADDITIVES IN BIODIESEL FOR ENHANCING THE PERFORMANCE OF CI ENGINE

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ARTICLE HISTORY

Received: 11.07.2022 Revised: 26.07.2022 Accepted: 28.07.2022

ABSTRACT

The present investigation aimed at the effects of addition of titanium dioxide nanoparticles with *Pongamia pinnata* biodiesel blends in a diesel engine as nanocatalysts, to overcome few disadvantages of biodiesel. Titanium dioxide nanoparticles were biosynthesized using neem leaf extract and were dispersed into the biodiesel blends using ultrasonicator. The proportion of doping of nanocatalysts with biodiesel blends was optimized. It was observed that brake power and brake thermal efficiency of the CI engine increased by 7.83 % and 4.2 % respectively, for B20$TiO_2$50 as compared to B20 whereas, CO and CO_2 emissions decreased.

KEYWORDS: Bio-synthesis, Brake power, Emission, Neem leaf extract, Titanium dioxide nanoparticles

Citation

Vidya Kulkarni, Vijayakumar Palled*, Prakash, K.V, Devanand Maski, Sharanagouda Hiregoudar and Sushilendra. Biosynthesis of titanium dioxide nanoparticles and their application as additives in biodiesel for enhancing the performance of CI engine. Scientist. 2022; 1(2): 448-462. DOI: https://doi.org/10.5281/zenodo.6924075

INTRODUCTION

India, the world's third largest oil consumer, currently buys over 80 per cent of its supplies and its crude oil consumption exceeded 4 million barrels per day. According to OPEC forecast, this will rise by as much as 5.8 million barrels per day by 2040 [1]. The increasing industrialization and modernization of the world has led to the increased demand of petroleum products, which entailed for alternative fuels produced from materials available within the country. Bio-diesel has become more attractive because of its environmental benefits, non-toxic and low cost. Bio-diesel production from edible seeds is not preferred in India as it imports 46% of edible oil to meet out the demand, therefore non-edible oils such as pongamia, jatropa, madhuca indica etc. are preferred over them which can be substituted either fully or partially with diesel oil. Modern diesel engine capability has forwarded to the point where the disadvantages of bio-fuel usage are eliminated. Recent diesel engines produce less noise, vibrations or smoke and they are additional fuel efficient than older model engines, biodiesel fuels achieve just as well as regular diesel fuels [2]. Some recent studies on the diesel fuel reformulation have been conducted on adding the possible metal nanoparticles. Metal and metal oxide nanoparticles have high surface to volume ratio which is responsible for their fascinating properties such as antimicrobial, magnetic, electronic and catalytic activity [3, 4]. Generally, properties of nanoparticles depend on size, shape, composition, morphology and crystalline phase. Among the various metal oxide nanoparticles, titanium dioxide nanoparticles have wide applications in air and water purification, DSSC due to their potential oxidation strength, high photo stability and non-toxicity [5].

The nanoparticles were synthesized by various physical, chemical and biological methods among which biological synthesis of nanoparticles is gaining prominence due to its eco-friendly products, biocompatibility and economic viability in the long run and also to avoid its adverse effects [6]. There are many studies on performance of CI engines using biodiesel in different proportions suggesting the impracticality of 100 % substitution of vegetable oil to the diesel but a blend of 20% vegetable oil and 80% diesel fuel may successful [7, 8]. However, the studies on the use of biodiesel blends with nano additives are limited. Few studies have reported that with the addition of nanoparticles with biodiesel, the performance of CI engine was improved [9, 10]. Considering the above facts in the view the present study aimed at the biosynthesis and characterization of titanium dioxide nanoparticles and analyze their effects with the biodiesel blend at different load conditions on performance of CI engine.

MATERIALS AND METHODS

Preparation of *Azaridachta indica* leaf extract

A. indica leaves were thoroughly washed using distilled water to remove dirt and washed leaves were shade dried at room temperature for 15 days at normal atmospheric conditions. Dried leaves were cut into fine pieces, grinded to get the finest powder passed through a 100 mesh sieve (150 µm). Ten grams of the dried leaves were mixed with 100 mL of ethanol and extracted under reflux condition at 50°C using soxlet apparatus (M/s Tarsons, 6090, Kolkata, India). After one hour, ethanolic leaf extract was obtained by filtering the mixture through Whatman No. 1 filter paper. The filtrate was stored at 4ºC for further experiments [11].

Biosynthesis of titanium dioxide nanoparticles using neem (*Azaridachta indica*) leaf extract.

Titanium dioxide nanoparticles were synthesized by dissolving, 0.5 M titanium isopropoxide in the mixture of 10 mL of ethanol and 40 mL of distilled water and homogenized for 15 min using homogenizer (M/s Tarsons, 6090, Kolkata, India). Ten mL of ethanolic leaf extract is added to 50 mL of homogenized mixture. The mixture was heated for 24 hours using magnetic stirrer (M/s Tarsons, 6090, Kolkata, India) at 50 ºC until colour was changed. Upon heating, the chemical reaction took place resulted in colour change in the reactants from white to brown and the mixture was taken off from the magnetic stirrer and cooled. The appearance of brown colour indicated the formation of titanium dioxide nanoparticles. The formed titanium dioxide nanoparticles were acquired by centrifugation at 5000 rpm for 15 minutes using high speed centrifuge (M/s Tarsons, 6090, Kolkata, India). Separated titanium dioxide nanoparticles were dried to calcination using muffle furnace at 550 ºC for five hours. The calcinated titanium dioxide nanopowder was used for further analytical analysis [12]. The biosynthesis of nanoparticles is presented in Plate. 1.

Characterization of biosynthesized titanium dioxide nanoparticles

Standard and biologically synthesized titanium dioxide nanoparticles were characterized using various instruments *viz*., Zetasizer used as dynamic light scattering apparatus to measure average particle size, UV-Visible Spectrophotometer refers to absorption spectrophotometer in the ultra-violet and visible spectral region of the electromagnetic spectrum. An optical spectrophotometer records the wavelengths at which absorption occurs, together with the degree of absorption at each wavelength The resulting spectrum was presented as a graph of absorbance (A) versus wavelength (λ), Scanning Electron Microscope (SEM) for morphological features of TiO_2 nanoparticles was obtained by scanning it with a high energy beam of electrons in vaccum chamber, X-Ray Diffraction (XRD analysis) is a unique method in determination of crystallinity of a compound and Atomic force microscope provides a 3D profile of the surface on a nanoparticles by measuring forces between a sharp probe (< 10 nm) and surface at very short distance. The biosynthesized nanoparticles were dispersed in the biodiesel blend of B20 using ultrasonicator with the mass fraction of 30, 40 and 50 ppm. The physico-chemical properties of modified fuel *viz,* Specific gravity, Kinematic viscosity, Calorific value, Flash point, Fire point, Pour point, Cloud point, Carbon residue, Free fatty acids and Acid value were determined using ASTM standards [13].

Engine test rig

An experimental setup (Plate 2) was used and the tests were conducted to study the various performance characteristics of CI engine using high speed diesel (HSD) and B20 biodiesel blends as a test fuels. The set up consists of single cylinder, four strokes, vertical cylinder and water cooled diesel engine was selected for the study. The engine is coupled to the rope brake dynamometer to measure the power. The specifications of engine are presented in Table 1. The engine was tested at three loads setting viz. 25, 50 and 75 % of rated load. The engine was loaded by adding different weights on the hanger till effective load obtained was equivalent to calculated effective load for a particular load setting. The dynamometer loads settings were adjusted to achieve the load on break drum. The test confirmed with IS 10000 – 1980 part VIII (Performance test). Reading was recorded after engine stabilizes in terms of load, speed and temperature.

During each testing, the engine operating temperature and lubricating oil pressure were maintained as recommended by the manufacturers. During each load, the various observations such as various temperatures, engine speed, fuel consumption, U-tube manometer reading, exhaust gas profile etc. were recorded to calculate various performance parameters.

Measurement of engine speed

The speed of engine was measured by using a contact type tachometer. Probe of the tachometer was in direct contact with the centre point of the mounted drum of dynamometer and the readings are displayed in revolution per minute (rpm). The speed was recorded manually by readings shown in the dial.

Brake power

The mechanical power output of the engine called as brake power, which represents the total power available for doing the useful work and was calculated by using formula (Hosseini *et al.*, 2017).

$$B.P. = \frac{2 \times \Pi \times N \times W \times R \times 9.81}{60 \times 1000}$$

Where,

B.P. – Brake power, kW

N – Engine speed, rpm

W – Brake load, kg

R – Radius of the brake drum, m

Brake thermal efficiency

The brake thermal efficiency was calculated by the brake power of the engine and heat supplied by the fuel [14].

$$\text{Brake thermal efficiency (\%)} = \frac{\text{Brake Power}}{\text{Heat value}} \times 100$$

Emission characteristics of an engine

The emission of exhaust gases like carbon monoxide (CO in ppm) and carbon dioxide (CO_2 in %) during operation of the engine with high speed diesel and biodiesel with nanoadditives were measured using a KM900 plus analyzer. The KM900 plus analyzer consists of a probe which can be directly inserted into the exhaust gas outlet. The gas analyzer directly indicates the values of the constituent gases and stores in the memory provided.

RESULTS AND DISCUSSION

Particle size analysis

The characterization of standard and biosynthesized Titanium dioxide nanoparticles was done in terms of average particle diameter from the intensity distribution analysis by using zetasizer. The size of standard and biosynthesized titanium dioxide nanoparticles was 99 nm and 56 nm, respectively (Fig 1).

Absorbance analysis

The UV-Visible absorption spectra of standard titanium dioxide nanoparticles exhibited characteristic Surface Plasmon resonance (SPR) band centered at wavelength of 358 nm and absorbance of 1.10. Whereas, for biosynthesized Titanium dioxide nanoparticles, SPR band cantered at wavelength of 280 nm and absorbance of 4.12.

Surface morphology analysis

The clear magnified (8.28 KX) SEM image at the accelerating voltage of 10.00 kV with working distance of 9.50 mm, showed that, uniformly distributed standard and biosynthesized titanium dioxide nanoparticles were in spherical shape (Fig 2).

Phase identification

The crystalline nature of titanium dioxide nanoparticles was confirmed from the X-ray diffraction analysis. Fig. 3 shows typical XRD pattern of the standard and biosynthesized titanium dioxide nanoparticles. XRD pattern showed five distinct diffraction peaks at 25.3°, 37.7°, 48.7°, 54.0° and 62.7° that were corresponding to 101, 004, 200, 105 and 204 reflection planes of biosynthesized titanium dioxide nanoparticles, respectively. The highest peak was observed at 25.3º (101) reflection. The XRD study confirmed that, the resultant nanoparticles were face centered tetrahedral in nature with anatase crystalline structure.

Surface analysis

Surface strength of titanium dioxide nanoparticles were studied using atomic force microscope (AFM). AFM micrographs with a scanning area of 10 × 10 µm of titanium dioxide nanoparticles in 2D and 3D images of the standard and biosynthesized titanium dioxide nanoparticles sample (Fig 4) showed spherical particles with different sizes. Height and width of standard titanium dioxide were 79 nm and 157 nm, respectively whereas for biosynthesized titanium dioxide nanoparticles were 2.45 and 2.20 µm were recorded, respectively.

Physical properties of biodiesel blends with nano additives

Table 2 shows the physical properties of fuel, the physical properties determined were specific gravity, Kinematic Viscosity, Calorific value, flash point, fire point, cloud point, pour point, carbon residue, free fatty acids and acid value. The specific gravity was found increasing with the increase in the proportion of nanoparticles which may be due to added weight to its density. Kinematic viscosity also increased with the increase in the proportion of nanoparticles may be due to increase in specific gravity as weight of nanoparticles also contributed to its density. The minimum and maximum viscosity values of 2.40 cSt and 3.40 cSt was recorded for biodiesel blends with nano additives of B10$TiO_2$30 and B40$TiO_2$50, respectively and Calorific value increased with the increase in the proportion of nanoparticles in the biodiesel blends, it may be due increase in the volatility of fuel.

Performance of CI engine with biodiesel blends with nano additives

A 3.75 kW, stationary, constant speed, single cylinder diesel engine was evaluated using diesel, bio-diesel (B20) and bio-diesel with nano additives under different load conditions and the results of performance and emission characteristics of engine are reported. The selected engine was evaluated in terms of brake power, brake thermal efficiency, biodiesel blend and biodiesel blend with nano additives, at constant speed and varying load conditions and compared with diesel.

Brake power

The brake power developed at different loads using diesel, biodiesel blend (B20) and biodiesel blends with nano additives was calculated and results are presented in Table 3 and Fig 5. From Table 3 it was observed that the Brake power generated from B20 was almost identical to the Brake power generated from Diesel which signifies its substitution. Among the different brake powers obtained for different proportion of nanoparticles, the brake power was highest for B20$TiO_2$50 at 75% load with the per cent increase of 7.83 % over B20. Brake power generated for B20 and B20 with different ppm of nanoparticles is shown in Fig.5. It was observed that the brake power increased with the increase in per cent of load, and increase in the proportion of nanoparticles. This may be due to the fact that addition of nanoparticles increased the surface to volume ratio that caused more amount of fuel to react with air which led to improve the performance of the engine [15].

Brake thermal efficiency

The brake thermal efficiency of the engine using diesel, biodiesel blend and biodiesel blend with three different proportions of nanoparticles was calculated at different load conditions and presented in Table 4 and Fig. 6. It was inferred from the Table 4 that, the Brake thermal efficiency of B20 was found increasing with the increase in the percentage of load and was on par with the power generated from the Diesel. It was also observed that among the different proportions, the brake thermal efficiency was found maximum (30.54%) for B20$TiO_2$50 at 75% load with the average increase of 4.2% when compared with B20 alone. (Fig. 6). The similar findings of brake thermal efficiency were reported for biodiesel blend with 40 ppm of titanium dioxide nanoparticles was 3.1% more when compared with biodiesel blend of B20 [12]. Brake thermal efficiency for B20 and B20 with different ppm of nanoparticles is shown in Fig.6. It was observed that the thermal efficiency increased with the increase in the percent of load and with the increase in the proportion of nanoparticles. This may be due to improved combustion, atomization and rapid evaporation of the nanoparticles that dispersed the test fuel resulting in better air fuel mixing, which allows more surface area of fuel to react with oxygen molecule [10].

CO emission

The CO emission of engine for diesel, biodiesel blend and modified fuel was analyzed and presented in Table 5 and Fig. 7. The CO emission of B20 was similar to that of emission obtained from the diesel and was found decreasing with the increase in the percentage of load. (Table 5) Decreasing trend was observed with the increase in the percent of load and proportion of nanoparticles which may be due to addition of nanoparticles shortened the ignition delay period, improved the air-fuel mixing ratio and higher carbon combustion activation which lead to complete combustion [8]. The CO emission found minimum for B20$TiO_2$50 with an average increase of 19% when compared with B20 (Fig.7). The CO emission was reduced by 23% for biodiesel blend with 40 ppm titanium dioxide nanoparticles when compared with B20 [12]. Fig 7 shows the CO emission for B20 and B20 with different proportions of nanoparticles.

CO_2 emission

The CO_2 emission of engine for diesel, biodiesel blend and modified fuel was analyzed and presented in Table 6 and Fig. 8. From Table 6 the CO_2 emission of B20 was almost nearer to the emission obtained by diesel. It was observed that CO_2 emission showed increasing trend with increase in the proportion of titanium dioxide nanoparticles which may be due to increase in the amount of oxygen molecule resulting in more amount of CO to combine with O_2 to form CO_2. (Fig. 8) Similar findings were reported for biodiesel blends containing aluminium oxide nanoparticles. The CO_2 emission for B20 and B20 with different proportion of nanoparticles is shown in Fig. 8.

Statistical analysis

The data obtained were analyzed using factorial design in the Design Expert software which is manifested in Table 7. The vales of mean, Standard Deviation and Co-efficient of variation for brake power were 1.8, 0.75 and 40.46 % respectively whereas for Brake thermal efficiency it was 23.71, 4.97 and 20.95 % respectively. The emission characteristics analyzed the values of mean standard deviation and co-efficient of variation for CO emission were 471.83, 71.98 and 15.26 % respectively and for CO_2 it was observed as 2.38, 1.28 and 53.93 respectively.

CONCLUSION

The Nanoparticles were synthesized biologically and characterized using neem leaf extract. The performance of diesel, biodiesel and biodiesel blend with nano additives in 3.75 kW diesel engines was evaluated for brake power, brake thermal efficiency. CO and CO_2 emission were also analyzed. Based on the results and discussions, the following conclusions were drawn.

- Green synthesis of nanoparticles resulted in the size of 56.13 nm when analyzed using Zetasizer.

- XRD confirmed the anatase crystalline structure of nanoparticles, SEM analysed revealed that the synthesized nanoparticles were spherical in shape.
- AFM analysis studied the morphology of synthesized nanoparticles.
- As load and percentage of biodiesel increased brake power was also increased. Brake power was increased by 7.83% for B20$TiO_2$50 when compared with B20 at 75% load.
- Brake thermal efficiency increased with the increase in the load. It was found that 4.2% more efficient for B20$TiO_2$50 when compared with B20.
- CO emission was decreased with the increase in the percentage of biodiesel blend and decreased with the increase in the proportion of nano additives in biodiesel blend.
- CO_2 emission decreased with increase in the per cent of biodiesel blend whereas increased with the increase in load. It decreased by 3% for B20$TiO_2$50 when compared with B20.

ACKNOWLEDGEMENT

We would like to thanks to Scientist R Academy, Bangalore India.

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Table 1 Specifications of the engine selected

Specifications	Remarks
Make	M/s. Rocket engineering, Kolhapur, Maharashtra state
Description	Four stroke , single cylinder engine
Fuel used	Diesel
Bore	80 mm
Stroke	110 mm
Piston displacement	50.272 cm^3
Speed	1500 rpm
Rated load	12.90 kg
Compression ratio	16:1
Output rated power	3.75 kW (5 hp)
Cooling system	Jacket type water cooling system
Lubrication system	Forced feed type

Table 3 Brake power at different loads for diesel (B0) and biodiesel blend (B20)

Loads, %	Blends	
	B0	B20
25	0.98	0.9
50	1.94	1.78
75	2.9	2.68

Table 4 Brake thermal efficiency at different loads for diesel (B0) and biodiesel blend (B20)

Loads,	Blends	
	B0	B20

%		
25	17.54	17.38
50	25.5	23.39
75	30.6	28.50

Table 5 CO emission at different loads for diesel (B0) and biodiesel blend (B20)		
Loads, %	**Blends**	
	B0	**B20**
25	690	680
50	590	520
75	510	480

Table 6 CO_2 emission of engine at different loads f diesel (B0) and biodiesel blend (B20)		
Loads, %	**Blends**	
	B0	**B20**
25	1.2	1.0
50	2	2.0
75	3.4	4.0

Dried leaves

Aqueous leaf extract

Reduced TiO_2 nanoparticles

Oven dried nanoparticles

Calcinated TiO_2 nanoparticles

Plate 1: Biosynthesis of titanium dioxide nanoparticles

Table. 7 The data for SD and CV for Brake Power, Brake Thermal Efficiency, CO Emission and CO_2 Emission of B20 as compared to B0

Parameters/ Attributes	Mean	Standard Deviation	Co-efficient of variation
Brake Power	1.8	0.75	40.46
Brake Thermal efficiency	23.71	4.97	20.95
CO Emission	471.83	71.98	15.26
CO_2 Emission	2.38	1.28	53.93

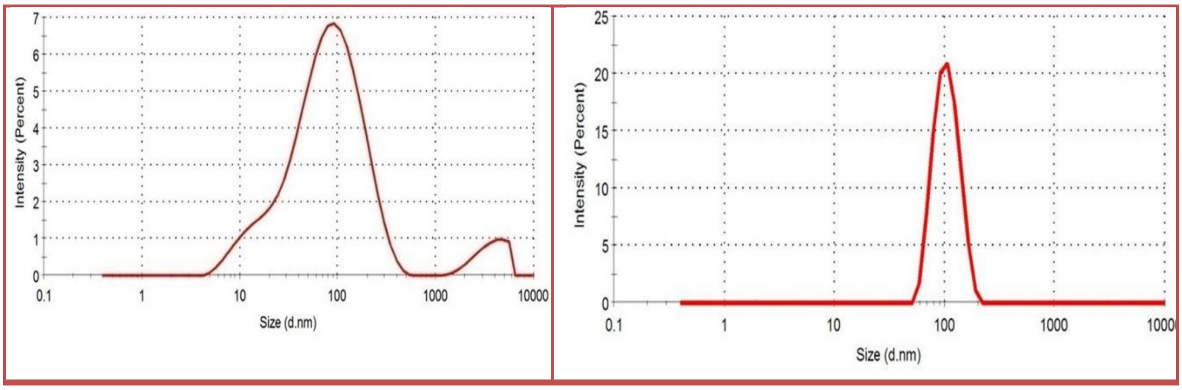

Figure 1: Size distribution by intensity of standard and biosynthesized titanium dioxide nanoparticles

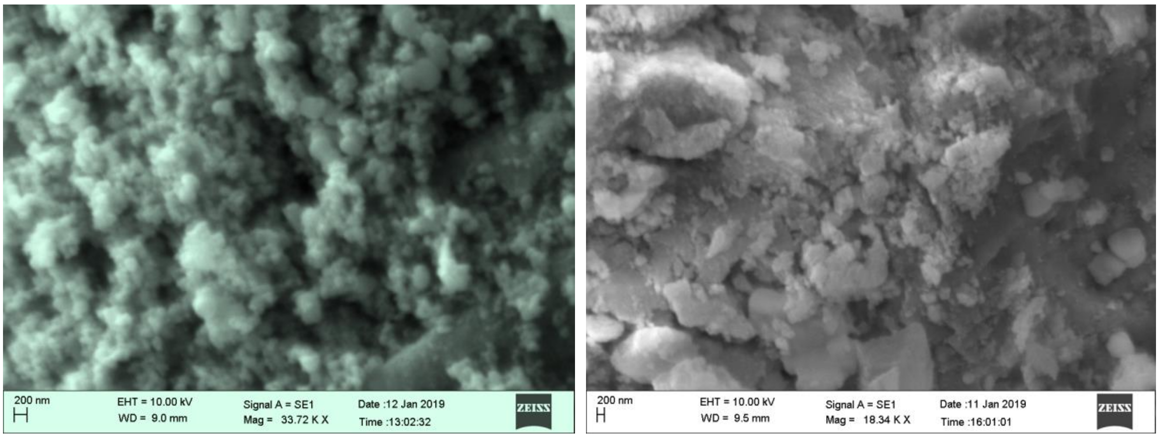

Figure 2: SEM image of standard and biosynthesized titanium dioxide nanoparticles

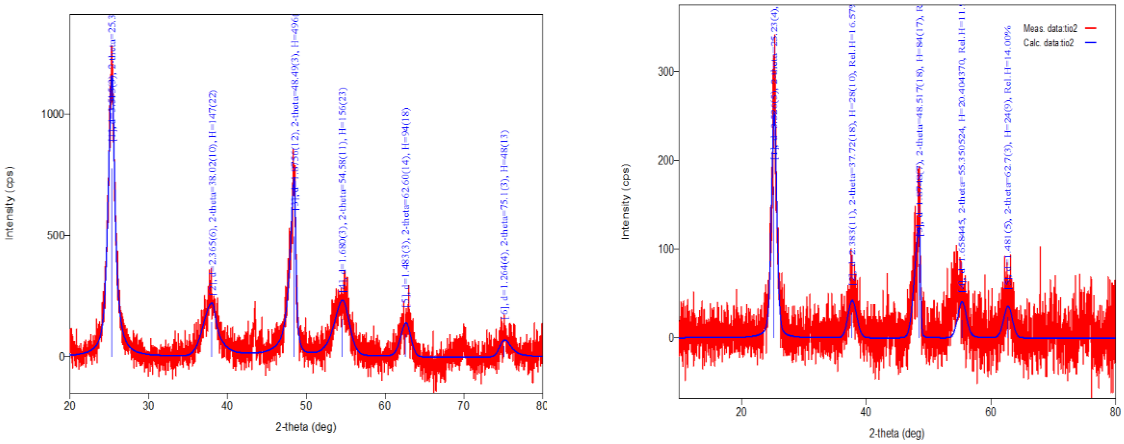

Figure 3: XRD image of standard and biosynthesized titanium dioxide nanoparticles

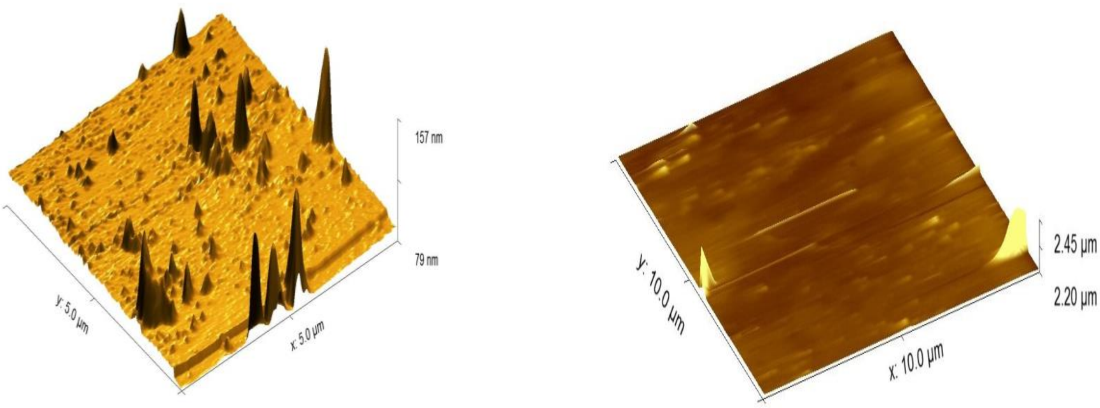

Figure 4: AFM image and profile of standard and biosynthesized titanium dioxide nanoparticles

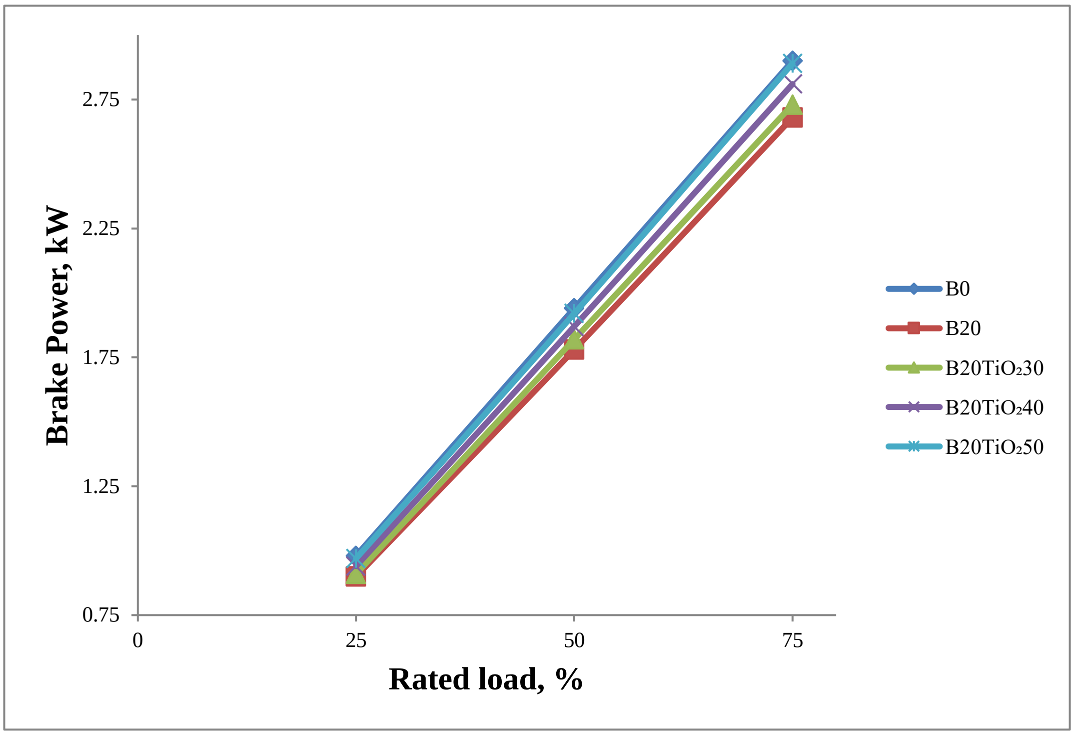

Figure 5: Brake power of engine for biodiesel blend of B20 with titanium dioxide nanoparticles

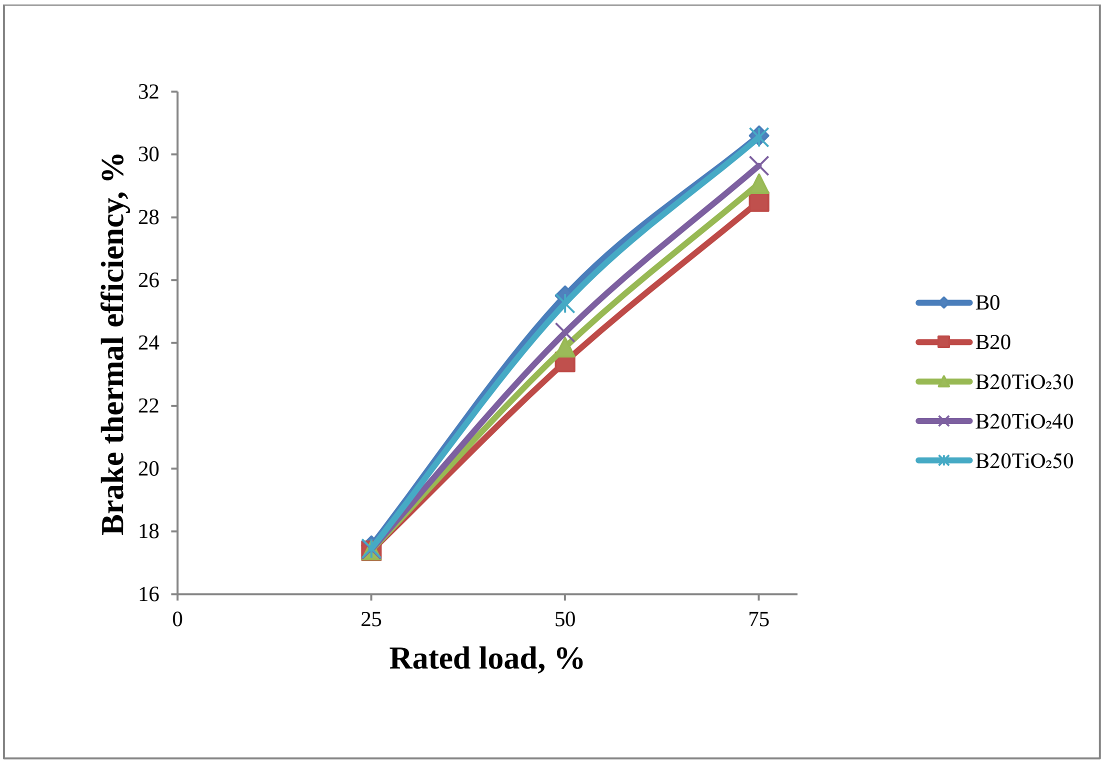

Figure 6: Brake thermal efficiency of engine for biodiesel blend of B20 with titanium dioxide nanoparticles

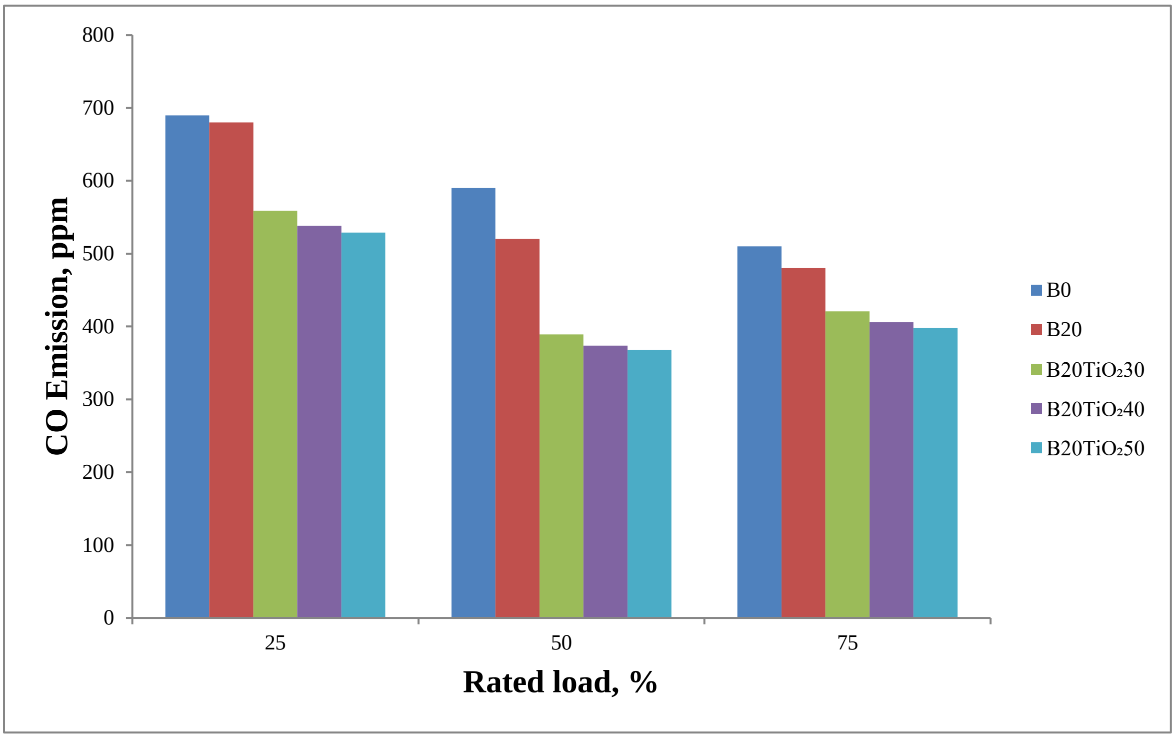

Figure 7: CO emission of engine for biodiesel blend of B20 with titanium dioxide nanoparticles

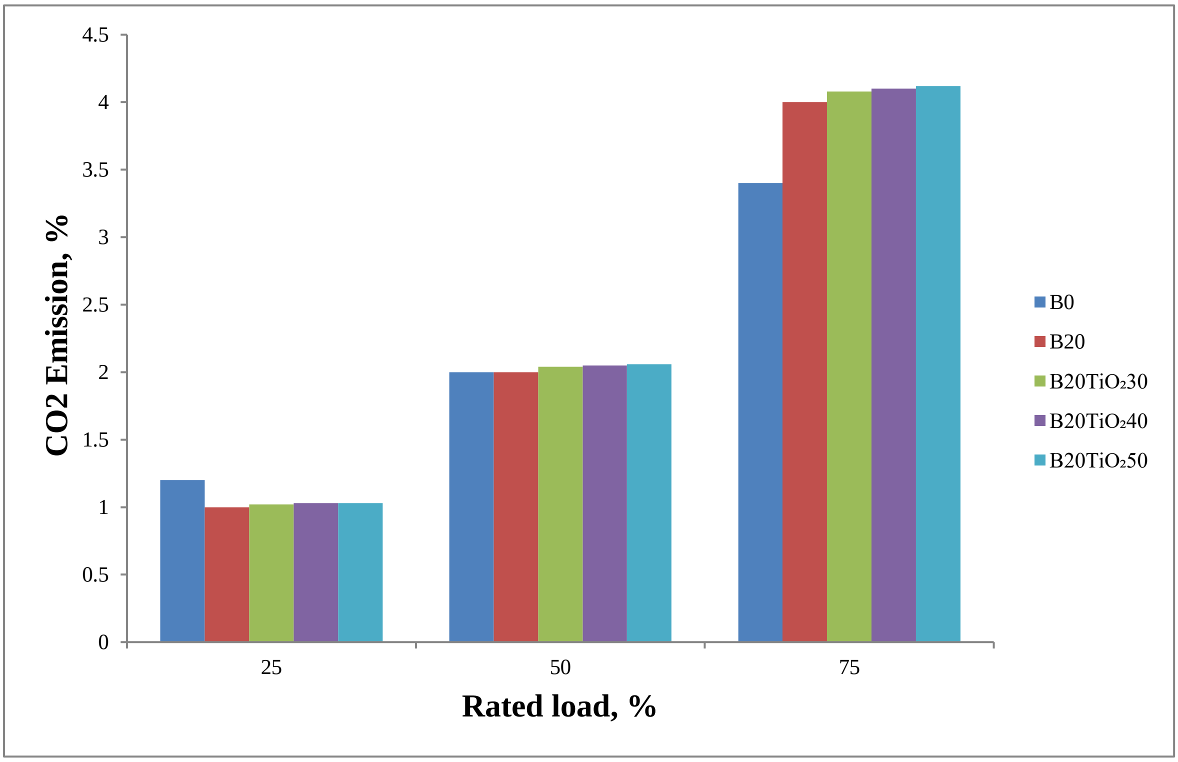

Fig.8 CO_2 emission of engine for biodiesel blend of B20 with titanium dioxide nanoparticles