

# Synthesis and characterization of Titanium dioxide nanoparticles against two insect pests

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**Abstract:** Nanosciences and biotechnology are nm scale investigation of incredibly small things. Specific and mixed-age *Tribolium castaneum* and *Trogoderma granarium* have been reported from the Faisalabad commodity market. The population had been acclimatized to the laboratory for each of the two insects. Pupa of the same age was collected during insect rearing and in separate plastic jars for adult emergence (2 weeks) to raise the homogenous population. Biosynthesis of titanium-particles was performed according to the normal protocol after the extraction of plant materials. Toxicity Bioassays were performed through three plant extract amounts (5, 10 and 15 %) (for both of the basic plant oils as well as nano-particles). Mortality results were reported after 24, 48 and 72 hours of care. *Tribolium castaneum* has the highest mortality rate (15.10 percent) and the lowest mortality rate (46.12 percent). The highest mortality rates for TiO<sub>2</sub> nanoparticles were 40.40 and 33.68 % respectively. Repellency bioassay was done by area preference method (TiO<sub>2</sub>) have 86.10 %, 72.64% against *Tribolium castaneum* and the *Trogoderma granarium*. Data of all the bioassays were analyzed by factorial under CRD statistical design.

**Keywords:** Titanium dioxide, Nanoparticles, Pests, FTIR, UV

## Introduction

Nanosciences is a developing and technologically evolved process that can encompass the fundamental elements and can recognize and advanced the evolving process of processing of materials, which have one component. This can regulate individual molecules and atoms. This is one of the most powerful innovations from the last decades (Hao *et al.*, 2011). Nanoparticles (Nanopowder, Nano ring, Nanocrystal) are the Nanoscience small building block (Ramezani and Mansoori, 2007). It also used as a biomedical, optical and electronic field. The nanoparticle has been work very successfully in the field of medicine, environmental sciences and food processing (Peng *et al.*, 2012). Owing to its modern use such as environmental protection, data management, genetics, beauty products, medicines owing to their electronic, physical and magnetic properties, nanoparticles have recently been commercial(Dagdeviren *et al.*, 2014). In fact, the biosynthesis of nanoparticles is environmentally sustainable, because it can shape the toxic, costly chemicals without the use of them (Trindade *et al.*, 2001)This has also been used as a highly conductive and semiconductor, medical equipment, sensor insulation, catalytic agents and as a pesticide as well. Nanocarriers are designed to reduce the amount of operation and delay the kinetics of agrochemicals releases (Pérez-de-Luque and Rubiales, 2009).

Store grains and their products are attacked by different insects which can cause huge losses (Ahmedani *et al.*, 2007). Crop storage accounted for about 9 percent of losses in industrialized countries and 20 percent in more emerging countries (Phillips and Throne, 2010). All stored grain insect pests most disreputable is red flour beetle *Tribolium castaneum* (Herbst) (Coleoptera: Tenebrionidae), which is a pervasive pest of the store commodities like wheat flour and crushed cereals, is also the cosmopolitan and most destructive insect pest (Lu *et al.*, 2010). *T. granarium* serious pest in the product which retained in the hot and dry conditions, with high relative humidity gives an acute effect on the population and building-up the sound grain pest (Ramzan and Chahal, 1986). Their adults are not dangerous but their larvae are most destructive but their adults are very harmless (Parashar, 2006). These larvae feed on sound grain and consume the entire kernel, leaving behind empty grains and husks. The quality of grains reduced due to frass and excreta in damage grains, it can create a negative effects on the atmosphere including infested grain have an adverse impact on human safety (Arain *et al.*, 2006).

*T. Granarium* stops grain from germinating because, as it assaults larvae during nutrition, it absorbs different nutrients (Jood and Kapoor, 1992). Other chemicals like pyrethroids are being also used for the control of stored grains insect-pests, but consistent use of these insecticides may lead to serious problems related to biochemical and hematological changes in human beings (Khan *et al.*, 2012). Conventional insecticides also pose a hazardous effect on non-target organisms including beneficial insects (Miller *et al.*, 2004). Due to the potential insecticidal properties botanical is the possible alternative source of pest control (Taheriniya and Behboodi, 2016). The effect of plant products showed insecticidal, repellent and antifeedant effects against insect pests (Ali *et al.*, 2017). In the past years, *T. castaneum* pest can be controlled by Polyethalyen glycol and diatomaceous earth but garlic essential oil can be act as a reducing agent (Yang *et al.*, 2009).

Nanomaterials may also prepare through biological System (easy, effective and environmentally-friendly, reducing the usage of harmful substances, using less resources and creating healthier materials and by-products) e.g. bacteria for Au, Ag, Zn and Fe NPs, yeasts for Ag and Pb NPs, plants for Au, Ag, Pd and Pt NPs (Taheriniya and Behboodi, 2016) Noble metal particles such as zinc oxide, magnesium oxide, titanium dioxide and silver are commonly employed in various fields owing to their antibacterial and antioxidant effects (Njagi *et al.*, 2011)

## **Preparation of Titanium oxide Nano-particles**

Firstly we can prepare the silver Nano particle like this way, Take the leaves powder from *R.communis*, *Jatropha curcus*, *Citrus paradise* purchased from the local market. Deionized water used in all experiments, I took 10g of *R. communis*, *Jatropha curcus*, and *Citrus paradise* extracts by using weight balance, which can be boiled in 100ml distilled water in 250ml conical flask. Then this extract was cooled at room temperature, Filter into the No. 1 filter paper of Whatman. It filtrate was an act of minimizing and stabilization agent for the preparation of nanoparticles of titanium oxide. Ammonium solution will be added to Titanium oxide (solution followed by the addition of plant material extract 1-10ml) as described in (Mirjalili *et al.*, 2017).

### **1. Bioassay of TiO<sub>2</sub> nanoparticles**

For the preparation of titanium nanoparticles, we can use the green synthesis method. 10ml of plant oil from stock solution boiled in 50 ml double distilled water for 30 minutes at 90°C. After this; through the use of the Whattman filter paper, we can filter this extract. We can prepare the 1mM solution of the TiO<sub>2</sub> aqueous solution which can boil at 25°C for 2 hours. Add 10ml of aqueous plant extract into the 20 ml of 1mM TiO<sub>2</sub> solution at 25°C heated with 200rpm stirring for 4hours. After 4 hours, the colors of the

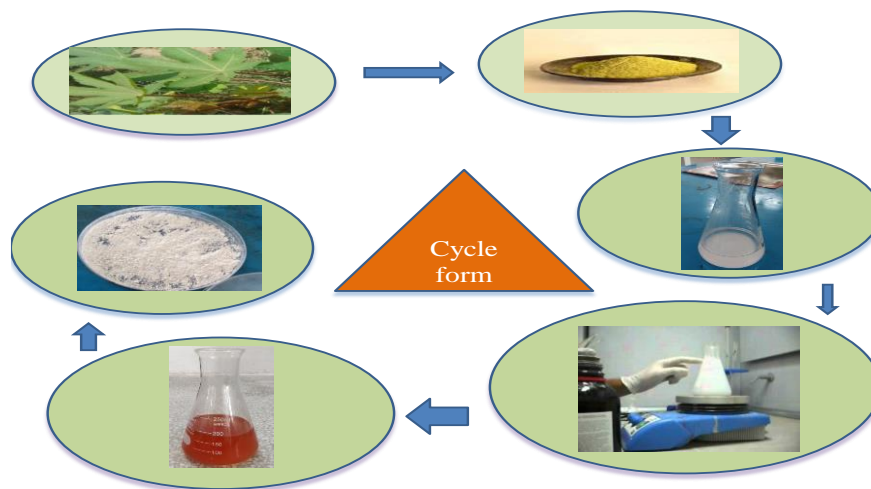
solution will be changed into lite green and then converted into colorless form. Then we can put the extract for 24 hours in oven for drying at 80°C. Then these precipitates were drying and stored for further analysis (Dobrucka, 2017).



**Figure 1.1** (Titanium-Dioxide Dried nano-composites)

**Table 1:** Change in solution color when forming Titanium dioxide nanoparticles using *Jatropha curcus*, *Citrus paradise*, and *R. communis* herb extracts of *Communis*.

Solution	Before Reduction	After Reduction	Color intensity	Time
<i>Jatropha curcus</i>	Dark Yellow			
1mM solution of TiO <sub>2</sub>	Transparent	Yellow	+	Immediately
		Pale Yellow	++	After 7 hours
		Off white	+++	After 24 hours
<i>Citrus paradise</i>	Lite Yellow			
1mM solution of TiO <sub>2</sub>	Transparent	Green	+	Immediately
		Yellowish	++	After 7 hours
		Pale yellow white	+++	After 24 hours
<i>R. communis</i>				
1mM solution of TiO <sub>2</sub>	Transparent	Yellow	+	Immediately
		Pale Yellow	++	After 7 hours
		Off white	+++	After 24 hours



**Figure 1.2:** Sketch Explanation

## CHARACTERIZATION OF NANOPARTICLES

We will estimate the optimum output of silver nanoparticles through absorption spectra. Using a UV-visible spectrophotometer (HITACHI, Model U-2800) we will detect the wavelength of SNP; deionized water serves as a blank solution. The ultimate top point of silver nanoparticle will be in an air-dried shape and atomic-force-microscopy (Model-Nanosurf easy scan 2 AFM, Switzerland) as defined in (Kasthuri *et al.*, 2009) will be able to classify it.

## TOXICITY BIOASSAY

Three herb extracts can be dissolved in the stock solution by using four solvents, their concentration is (15, 10 and 5 %). The concentration is to be hands-on smashed seeds, shook for better concentration delivery. This will allow air to be dried and placed into small plastic bottles. Both species, adults, will be released as defined in a controlled diet that includes jars (Rehman *et al.*, 2018).

### Repellency

The mean percentage of repellence was calculated by using a preferential approach. After the filter paper was divided into two parts, one half of each paper was prepared with the extracts from the plants and acetone. After some time, the solution would evaporate, so two halves were inserted and put in a Petri bowl. 20 *Tribolium castanum* and *Trogoderma granarium* adults were re-allocated to a filter paper that was screened in the middle. Repellency data were reported respectively after 24, 48 and 72 hours (Girshick *et al.*, 2015).

## Results and discussion

The studies have been repeated in thrice along with the control factor under different designs Fully Randomized Design (CRD) with all the various treatments. Plant extracts have been used at three different concentration levels, i.e. 5, 10 and 15 %, mortality results should be observed after 24, 48 and 72 hours of application of medication. At the same product type/rate of plant

extracts, we had independently performed the experiments on repellence and growth inhibition. The data were collected about larval inhibition, pupa; inhibition and adult inhibition at daily intervals, while the repellency data was reported at 24 hrs.

### WITH Titanium dioxide NANO-PARTICLES against *Tribolium castaneum*

#### 2. MORTALITY DATA AFTER EXPOSURE OF 24 HRS

To measure T-mortality. Castaneum, homogeneous adults were introduced into small plastic jars on a controlled diet. Adults were required to feed on controlled diets, and mortality data were reported. Wheat grains were used as diet and three concentrations of each plant extract were used viz., 5, 10 and 15%. Mortality data were recorded for 24, 48 and 72 h of the exposure period. Insects were held at  $30 \pm 2^{\circ}$  C and  $60 \pm 5$  per cent RH in incubators for mortality assessment. They repeated each operation and control three times.

Table 2.1 Reveals the variance analysis (ANOVA) of data on mean T percent mortality. Castaneum at various *Jatropha curcus*, *Citrus paradise*, and R concentrations. Commons.

Data showed that main effects, plants ( $F=4.66$ ;  $df=1$ ;  $p<0.05$ ) and concentration ( $F=11.10$   $df=2$ ;  $p<0.05$ ) were significant regarding mortality values of *T. castaneum* after an exposure period of 24 hours.

**Table 2.1** Analysis of variance (ANOVA) of the data concerning % mortality of *Tribolium castaneum* (Herbst) for Titanium dioxide-based nano-particles

S.O.V	DF	SS	MSS	F value
Plant	2	555.807	277.904	38.4000**
Concentration(Conc.)	2	480.691	240.345	33.2103**
Plant*Concentration	4	65.422	16.355	2.2600
Error	18	130.267	7.237	
Total	26	1232.187		

**Table 2.2** Comparison of the mean percentage mortality of *Tribolium castaneum* after exposure to different concentrations of plant extracts after 24 hrs

Concentrations (%)	Mean percentage of mortality $\pm$ SE
5	5.81 $\pm$ 1.49
10	14.18 $\pm$ 2.30
15	22.58 $\pm$ 4.77

Data in table 2.1 Represents the insecticidal effect of different concentrations of 3 different oil against *Tribolium castaneum*. The experimental data revealed that maximum mortality (22.58%) at 15% was recorded. The mean mortality was 14.18 per cent at 10 per cent concentration and 5.81 per cent death at 5 per cent plant extract concentration was observed. From this, it is inferred that mortality only increased with a rise in concentrations of the 3 different plant oil and shows also that concentration has a major impact on the mean percent mortality rate of *T. castaneum*.

**2.3 Comparison of the mean percentage mortality of *Tribolium castaneum* after exposure to different plant extracts after 24 hrs**

Concentrations (%)	Mean percentage of mortality ± SE
<b>P1</b>	6.67±1.44
<b>P2</b>	11.12±1.82
<b>P3</b>	24.80±4.48

Table 2.3. For percent mean mortality values of different plant extracts at different concentration levels showed that extracts of *R. communis* and *Jatropha curcus* gave mortality values 24.80 and 11.12%, correspondingly. While the least mortality 6.67 % was given by extract of *Citrus paradise*.

**2.4 Comparative mean percentage mortality of *Tribolium castaneum* after exposure to different concentrations of plant extracts after 24 hrs**

Plant extracts x Concentrations (%)	(%) Mean Mortality ± SE
<i>Citrus paradise</i> x 5	1.67±1.67 g
<i>Citrus paradise</i> x 10	8.34±1.67 efg
<i>Citrus paradise</i> x 15	10.00±0.00 def
<i>Jatropha curcus</i> x 5	5.00±0.00 fg
<i>Jatropha curcus</i> x 10	11.67±1.67 cd
<i>Jatropha curcus</i> x 15	16.67±1.67 bc
<i>Ricinus communis</i> x 5	10.77±1.68 de
<i>Ricinus communis</i> x 10	22.56±1.68 b
<i>Ricinus communis</i> x 15	41.07±1.68 a

Table 2.4 showed the interaction between different concentrations (5, 10 and 15%) and different exposure periods. Mean mortality of *T. castaneum* was given in percentage by the application of an extract of *Ricinus communis*, *Jatropha curcus*, *Citrus paradise* oil along with the standard error in table 2.4.

The mean comparison of percentage mortality values of *T. castaneum* at different concentrations of selected plant extract was highest at maximum concentration. Extract of *Ricinus communis* gave the highest mean mortality revealed that maximum mortality (41.07 %) at 15% was recorded. The mean mortality was 22.56% at 10% concentration and 10.78% mortality was detected at 5 percent of the plant extract concentration. Extract of *Jatropha curcus* gave the mean mortality revealed that maximum mortality (16.67%) at 15% was recorded. The mean mortality was 11.67% at 10% concentration and 5.00% mortality was observed at 5 per cent plant extract concentration. Extract of *Citrus paradise* gave the mean mortality revealed that maximum mortality (10.00%) at 15% was recorded. The mean mortality was 8.34% at 10% concentration and 1.67% mortality of the plant extracts was estimated at 5 percent. The result given showed substantial correlation between exposure time and concentration. From the results, we inferred that mortality values were rising slowly, with a rise in plant extract concentration.

### 3. MORTALITY DATA AFTER EXPOSURE OF 48 HRS

**Table 3.1** Analysis of variance (ANOVA) of the data concerning % mortality of *Tribolium castaneum* (Herbst) for Titanium dioxide-based nano-particles

S.O.V	DF	SS	MSS	F value
<b>Plant</b>	2	1608.555	804.278	123.0181**
<b>Concentration(Conc.)</b>	2	1265.198	632.599	96.7590**
<b>Plant*Concentration</b>	4	457.108	114.277	17.4792*
<b>Error</b>	18	117.682	6.538	
<b>Total</b>	26	3448.543		

**Table 3.2** Comparison of the mean percentage mortality of *Tribolium castaneum* after exposure to different concentrations of plant extracts after 48 hrs

Concentrations (%)	Mean percentage mortality ± SE
<b>5</b>	5.81±1.49
<b>10</b>	14.18±2.30
<b>15</b>	22.58±4.77

Data in table 3.1. Represents the insecticidal effect of different concentrations of 3 different oil against *Tribolium castaneum*. The experimental data revealed that maximum mortality (22.58%) at 15% was recorded. The mean mortality was 14.18 % at 10%

concentration and 5.81% mortality was observed at 5 per cent plant extract concentration. Through this, it is inferred that mortality only increased with a rise in concentrations of the 3 separate plant oil and indicates also that concentration has a major impact on the mean percent mortality rate of *T. Castaneum*.

**3.3 Comparison of the mean percentage mortality of *Tribolium castaneum* after exposure to different plant extracts after 48 hrs**

Concentrations (%)	Mean percentage of mortality ± SE
P1	6.67±1.44
P2	11.12±1.82
P3	24.80±4.48

Table 3.1 For percent mean mortality values of different plant extracts at different concentration levels showed that extracts of *R. communis* and *Jatropha curcus* gave mortality values 24.80 and 11.12%, correspondingly. While the least mortality 6.67 % was given by extract of *Citrus paradise*.

**3.4 Comparative mean percentage mortality of *Tribolium castaneum* after exposure to different concentrations of plant extracts after 48 hrs**

Plant extracts x Concentrations (%)	(%) Mean Mortality ± SE
<i>Citrus paradise</i> x 5	1.67±1.67 g
<i>Citrus paradise</i> x 10	8.34±1.67 efg
<i>Citrus paradise</i> x 15	10.00±0.00 def
<i>Jatropha curcus</i> x 5	5.00±0.00 fg
<i>Jatropha curcus</i> x 10	11.67±1.67 cd
<i>Jatropha curcus</i> x 15	16.67±1.67 bc
<i>Ricinus communis</i> x 5	10.77±1.68 de
<i>Ricinus communis</i> x 10	22.56±1.68 b
<i>Ricinus communis</i> x 15	41.07±1.68 a

Table 3.4 showed the interaction between different concentrations (5, 10 and 15%) and different exposure periods. Mean mortality of *T. castaneum* was given in percentage by the application of an extract of *Ricinus communis*, *Jatropha curcus*, *Citrus paradise* oil along with the standard error in table 3.4



The mean comparison of percentage mortality values of *T. castaneum* at different concentrations of selected plant extract was highest at maximum concentration. Extract of *Ricinus communis* gave the highest mean mortality revealed that maximum mortality (41.07 %) at 15% was recorded. The mean mortality was 22.56% at 10% concentration and 10.78% mortality concentration of plant extracts was observed at 5 per cent. Extract of *Jatropha curcus* gave the mean mortality revealed that maximum mortality (16.67%) at 15% was recorded. The mean mortality was 11.67% at 10% concentration and 5.00% mortality the plant extract concentration was measured at 5 per cent. Extract of *Citrus paradise* gave the mean mortality revealed that maximum mortality (10.00%) at 15% was recorded. The mean mortality was 8.34% at 10% concentration and 1.67% mortality the concentration of plant extracts was measured at 5 per cent. The result given showed substantial correlation between exposure time and concentration. From the results, we concluded that mortality values were rising slowly, with a rise in plant extract concentration.

#### 4. MORTALITY DATA AFTER EXPOSURE OF 72 HRS

**Table 4.1** Analysis of variance (ANOVA) of the data concerning % mortality of *Tribolium castaneum* (Herbst) for Titanium dioxide-based nanoparticles

S.O.V	DF	SS	MSS	F value
<b>Plant</b>	2	967.452	483.726	94.106**
<b>Concentration</b>	2	1575.647	787.823	153.266**
<b>Plant*Concentration</b>	4	275.269	68.817	13.388*
<b>Error</b>	18	92.525	5.140	
<b>Total</b>	26			

**Table 4.2.** Comparison of the mean percentage mortality of *Tribolium castaneum* after exposure to different concentrations of plant extracts after 72 hrs

Concentrations (%)	Mean percentage of mortality ± SE
<b>5</b>	9.04± 0.87c
<b>10</b>	19.65± 2.41b
<b>15</b>	27.69± 3.45a

Data in table 4.1. Represents the insecticidal effect of different concentrations of 3 different oil against *Tribolium castaneum*. The experimental data revealed that maximum mortality (27.69%) at 15% was recorded. The mean mortality was 9.04 % at 5% concentration and 19.65% mortality was found at a plant extract concentration of 10 per cent. From this, it is concluded that Mortality increased even with a rise in concentrations of the three different plant oil and also indicates that concentration has a major impact on the mean percent mortality rate of *T. castaneum*.

**4.3 Comparison of the mean percentage mortality of *Tribolium castaneum* after exposure to different plant extracts after 72 hrs**

Concentrations (%)	Mean percentage of mortality ± SE
P1	11.89± 1.70c
P2	18.00± 2.29b
P3	26.49± 4.34a

Table 4.3. For percent mean mortality values of different plant extracts at different concentration levels showed that extracts of *R. communis* and *Jatropha curcus* gave mortality values 26.49 and 18.00%, corresponding. While the least mortality 11.89% was given by extract of *Citrus paradise*.

Comparative mean percentage mortality of *Tribolium castaneum* after exposure to different concentrations of plant extracts after 72hrs

Plant extracts x Concentrations (%)	(%) Mean Mortality ± SE
<i>Citrus paradise</i> x 5	6.334±1.34g
<i>Citrus paradise</i> x 10	12.00±1.15efg
<i>Citrus paradise</i> x 15	17.34±1.20def
<i>Jatropha curcus</i> x 5	9.67±0.34fg
<i>Jatropha curcus</i> x 10	19.00±0.57cd
<i>Jatropha curcus</i> x 15	25.34±0.67bc
<i>Ricinus communis</i> x 5	11.12±1.16de
<i>Ricinus communis</i> x 10	27.94±2.04b
<i>Ricinus communis</i> x 15	40.40±2.10a

Table 4.2. Showed the interaction between different concentrations (5, 10 and 15%) and different exposure periods. Mean mortality of *T. castaneum* was given in percentage by the application of an extract of *Ricinus communis*, *Jatropha curcus*, *Citrus paradise* oil along with the standard error in table 4.2.

The mean comparison of percentage mortality values of *T. castaneum* at different concentrations of selected plant extract was highest at maximum concentration. Extract of *Ricinus communis* gave the highest mean mortality revealed that maximum mortality (40.40%) at 15% was recorded. The mean mortality was 27.94% at 10% concentration and 11.12% mortality was observed at 5 per cent plant

extract concentration. Extract of *Jatropha curcus* gave the mean mortality revealed that maximum mortality (25.34%) at 15% was recorded. The mean mortality was 19.00% at 10% concentration and 9.67% mortality of the plant extracts was observed at 5 percent. Extract of *Citrus paradise* gave the mean mortality revealed that maximum mortality (17.34%) at 15% was recorded. The mean mortality was 12.00% at 10% concentration and 6.34% mortality was observed at 5 percent of the plant extract concentration. The result given showed substantial interaction between exposure time and concentration. From the analysis, we observed that mortality values were rising slowly, with a rise in plant extract concentration.

**WITH Titanium dioxide NANO-PARTICLES against *Trogoderma granarium***

**5. MORTALITY DATA AFTER EXPOSURE OF 24 HRS**

To evaluate the mortality of *T. granarium*, homogenous adults were released on the treated diet in small plastic jars. We found from the study that mortality values were increasing gradually, with a rise in concentration of plant extracts. Wheat grains were used as diet and three concentrations of each plant extract were used viz., 5, 10 and 15%. Mortality data were recorded for 24, 48 and 72 h of the exposure period. Insects were held at 30±2 oC and 60±5 per cent RH in incubators for mortality assessment. They repeated each treatment and control three times.

Table 5.1 reveals the analysis of variance (ANOVA) of data regarding the mean percentage mortality of *T. granarium* at different concentrations of *Jatropha curcus*, *Citrus paradise*, and *R. communis*. Data showed that main effects, plants (F=4.66; df=1: p<0.05) and concentration (F=11.10 df=;2 p<0.05) were significant regarding mortality values of *T. castaneum* after an exposure period of 24 hours.

**Table 5.1.** Analysis of variance (ANOVA) of the data concerning % mortality of *T. granarium* (Herbst) for Titanium dioxide-based nanoparticles

S.O.V	DF	SS	MSS	F value
Plant	2	871.817	435.909	114.508 **
Concentration	2	970.047	485.023	127.409 **
Plant*Concentration	4	180.138	45.035	11.830 *
Error	18	68.523	3.807	
Total	26	2090.525		

**Table 5.2** Comparison of the mean percentage mortality of *Trogoderma granarium* after exposure to different concentrations of plant extracts after 24 hrs

Concentrations (%)	Mean percentage of mortality ± SE
5	6.36±1.01
10	16.41±2.36

Concentrations (%)	Mean percentage of mortality ± SE
15	20.66±3.00

Data in table 5.1. Represents the insecticidal effect of different concentrations of 3 different oil against *Trogoderma granarium*. The experimental data revealed that maximum mortality (20.66%) at 15% was recorded. The mean mortality was 6.36% at 5% concentration and 16.41% mortality was observed at a concentration of 10 per cent of plant extracts. From this, it is inferred that mortality only increased with a rise in concentrations of the 3 different plant oil and shows also that concentration has a major impact on the mean percent mortality rate of *T. granarium*.

**5.3** Comparison of the mean percentage mortality of *Trogoderma granarium* after exposure to different plant extracts after 24 hrs

Concentrations (%)	Mean percentage of mortality ± SE
P1	7.23±1.12
P2	15.12±2.20
P3	21.09±3.29

Table 5.2. For percent mean mortality values of different plant extracts at different concentration levels showed that extracts of *R. communis* and *Jatropha curcus* gave mortality values 21.09 and 15.12%, corresponding. While the least mortality 7.23% was given by extract of *Citrus paradise*.

**5.4** Comparative mean percentage mortality of *Trogoderma granarium* after exposure to different concentrations of plant extracts after 24 hrs

Plant extracts x Concentrations (%)	(%) Mean Mortality ± SE
<i>Citrus paradise</i> x 5	3.34±1.67g
<i>Citrus paradise</i> x 10	8.34±0.34efg
<i>Citrus paradise</i> x 15	10.00±0.00def
<i>Jatropha curcus</i> x 5	7.00±1.00fg
<i>Jatropha curcus</i> x 10	17.00±1.52cd
<i>Jatropha curcus</i> x 15	21.34±0.89bc
<i>Ricinus communis</i> x 5	8.75±0.89de
<i>Ricinus communis</i> x 10	23.90±1.78b

Plant extracts x Concentrations (%)	(%) Mean Mortality ± SE
<i>Ricinus communis</i> x 15	30.63±0.67a

Table 5.5 showed the interaction between different concentrations (5, 10 and 15%) and different exposure periods. Mean mortality of *T. granarium* was given in percentage by the application of an extract of *Ricinus communis*, *Jatropha curcus*, *Citrus paradise* oil along with the standard error in table 5.5.

The mean comparison of percentage mortality values of *T. granarium* at different concentrations of selected plant extract was highest at maximum concentration. Extract of *Ricinus communis* gave the highest mean mortality revealed that maximum mortality (30.63%) at 15% was recorded. The mean mortality was 23.90% at 10% concentration and 8.75% mortality was observed at 5 per cent plant extract concentration. Extract of *Jatropha curcus* gave the mean mortality revealed that maximum mortality (21.34%) at 15% was recorded. The mean mortality was 17.00% at 10% concentration and 7.00% mortality concentration of plant extracts was observed at 5 per cent. Extract of *Citrus paradise* gave the mean mortality revealed that maximum mortality (10.00%) at 15% was recorded. The mean mortality was 8.34% at 10% concentration and 3.34% mortality was observed at 5% concentration of the plant extracts. The given outcome showed that the interaction of exposure time and concentration was significant. From the findings, we observed that mortality values were rising slowly, with a rise in plant extract concentration.

#### 6. MORTALITY DATA AFTER EXPOSURE OF 48 HRS

**Table 6.1** Analysis of variance (ANOVA) of the data concerning % mortality of *T. granarium* (Herbst) for Titanium dioxide-based nanoparticles

S.O.V	DF	SS	MSS	F value
<b>Plant</b>	2	580.743	290.371	80.454**
<b>Concentration</b>	2	717.708	358.854	99.429**
<b>Plant*Concentration</b>	4	159.210	39.802	11.028*
<b>Error</b>	18	64.964	3.609	
<b>Total</b>	26	1522.625		

**Table 6.2** Comparison of the mean percentage mortality of *Trogoderma granarium* after exposure to different concentrations of plant extracts after 48 hrs

Concentrations (%)	Mean percentage of mortality ± SE
5	5.14±0.72
10	13.40±1.92
15	17.53±2.65

Data in table 6.1. Represents the insecticidal effect of different concentrations of 3 different oil against *Trogoderma granarium*. The experimental data revealed that maximum mortality (17.53%) at 15% was recorded. The mean mortality was 5.14% at 5% concentration and 13.40% mortality was observed at 10% concentration of the plant extracts. From this, it is inferred that mortality only increased with a rise in concentrations of a 3 different plant oil and shows also that concentration has a major impact on the mean percent mortality rate of *T. granarium*.

**6.2** Comparison of the mean percentage mortality of *Trogoderma granarium* after exposure to different plant extracts after 48 hrs

Concentrations (%)	Mean percentage of mortality ± SE
P1	7.23±1.21
P2	10.56±1.55
P3	18.29±3.04

Table 6.3 For percent mean mortality values of different plant extracts at different concentration levels showed that extracts of *R. communis* and *Jatropha curcus* gave mortality values 18.29 and 10.56%, correspondingly. While the least mortality 7.23% was given by extract of *Citrus paradise*.

**6.4** Comparative mean percentage mortality of *Trogoderma granarium* after exposure to different concentrations of plant extracts after 48 hrs

Plant extracts x Concentrations (%)	(%) Mean Mortality ± SE
<i>Citrus paradise</i> x 5	3.34±1.67g
<i>Citrus paradise</i> x 10	8.34±1.67efg
<i>Citrus paradise</i> x 15	10.00±0.00def
<i>Jatropha curcus</i> x 5	5.00±0.00fg
<i>Jatropha curcus</i> x 10	11.67±1.67cd
<i>Jatropha curcus</i> x 15	15.00±0.00bc
<i>Ricinus communis</i> x 5	7.07±0.00de
<i>Ricinus communis</i> x 10	20.20±1.014b
<i>Ricinus communis</i> x 15	27.60±1.22a

Table 6.4 showed the interaction between different concentrations (5, 10 and 15%) and different exposure periods. Mean mortality of *T. granarium* was given in percentage by the application of an extract of *Ricinus communis*, *Jatropha curcus*, *Citrus paradise* oil along with the standard error in table 6.4.

The mean comparison of percentage mortality values of *T. granarium* at different concentrations of selected plant extract was highest at maximum concentration. Extract of *Ricinus communis* gave the highest mean mortality revealed that maximum mortality (27.60%) at 15% was recorded. The mean mortality was 20.20% at 10% concentration and 7.07% mortality concentration of plant extracts was observed at 5 per cent. Extract of *Jatropha curcus* gave the mean mortality revealed that maximum mortality (15.00%) at 15% was recorded. The mean mortality was 11.67% at 10% concentration and 5.00% mortality was observed at 5% concentration of the plant extracts. Extract of *Citrus paradise* gave the mean mortality revealed that maximum mortality (10.00%) at 15% was recorded. The mean mortality was 8.34% at 10% concentration and 3.34% mortality was observed at 5 per cent plant extract concentration. The result given showed substantial association between exposure time and concentration. From the findings, we estimated that mortality values were rising slowly, with a rise in plant extract concentration.

### 7. MORTALITY DATA AFTER EXPOSURE OF 72 HRS

**Table 7.1** Analysis of variance (ANOVA) of the data concerning % mortality of *T. granarium* (Herbst) for Titanium dioxide-based nanoparticles

S.O.V	DF	SS	MSS	F value
<b>Plant</b>	2	619.787	309.894	55.938**
<b>Concentration</b>	2	1246.537	623.268	112.504**
<b>Plant*Concentration</b>	4	186.387	46.597	8.411*
<b>Error</b>	18	99.719	5.540	
<b>Total</b>	26			

**Table 7.2** Comparison of the mean percentage mortality of *Trogoderma granarium* after exposure to different concentrations of plant extracts after 72 hrs

Concentrations (%)	Mean percentage of mortality ± SE
<b>5</b>	7.69±0.73
<b>10</b>	15.63±1.69
<b>15</b>	24.34±3.02

Data in table 7.1. Represents the insecticidal effect of different concentrations of 3 different oil against *Trogoderma granarium*. The experimental data revealed that maximum mortality (24.34%) at 15% was recorded. The mean mortality was 7.69% at 5% concentration and 15.63% mortality was observed at a concentration of 10 per cent of plant extracts. From this, it is inferred that

mortality only increased with a rise in concentrations of the 3 different plant oil and shows also that concentration has a major impact on the mean percent mortality rate of *T. granarium*.

**7.3 Comparison of the mean percentage mortality of *Trogoderma granarium* after exposure to different plant extracts after 72 hrs**

Concentrations (%)	Mean percentage of mortality ± SE
P1	9.67±1.32
P2	16.67±2.41
P3	21.34±3.72

Table 7.4 for percent mean mortality values of different plant extracts at different concentration levels showed that extracts of *R. communis* and *Jatropha curcus* gave mortality values 21.34 and 16.67%, correspondingly . While the least mortality 9.67% was given by extract of *Citrus paradise*.

**7.5 Comparative mean percentage mortality of *Trogoderma granarium* after exposure to different concentrations of plant extracts after 72 hrs**

Plant extracts x Concentrations (%)	(%) Mean Mortality ± SE
<i>Citrus paradise</i> x 5	5.00±0.00g
<i>Citrus paradise</i> x 10	10.00±0.00efg
<i>Citrus paradise</i> x 15	14.00±0.57def
<i>Jatropha curcus</i> x 5	9.00±0.00fg
<i>Jatropha curcus</i> x 10	15.67±0.34cd
<i>Jatropha curcus</i> x 15	25.34±1.34bc
<i>Ricinus communis</i> x 5	9.091±1.01de
<i>Ricinus communis</i> x 10	21.22±1.74b
<i>Ricinus communis</i> x 15	33.68±3.21a

Table 7.6. Showed the interaction between different concentrations (5, 10 and 15%) and different exposure periods. Mean mortality of *T. granarium* was given in percentage by the application of an extract of *Ricinus communis*, *Jatropha curcus*, *Citrus paradise* oil along with the standard error in table 7.6.

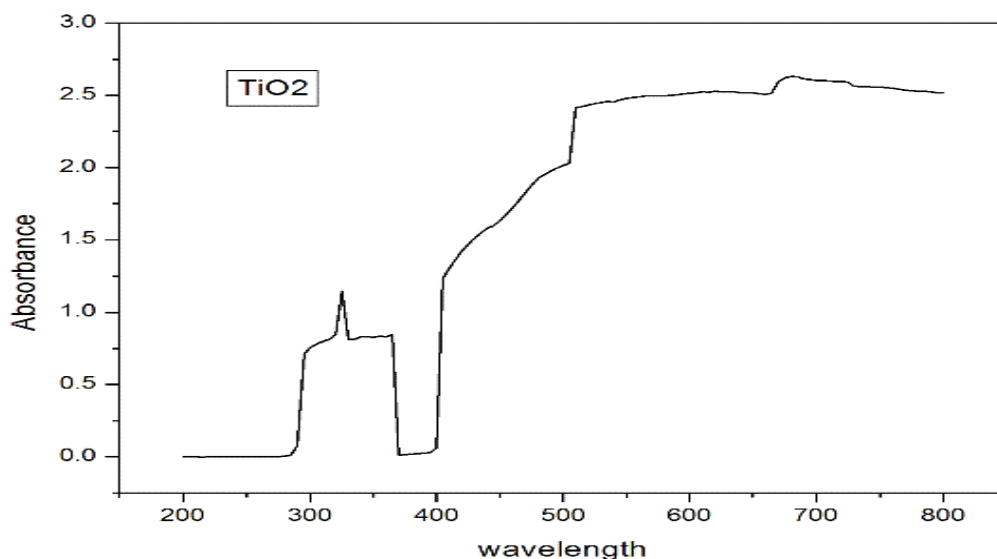


The mean comparison of percentage mortality values of *T. granarium* at different concentrations of selected plant extract was highest at maximum concentration. Extract of *Ricinus communis* gave the highest mean mortality revealed that maximum mortality (33.68%) at 15% was recorded. The mean mortality was 21.22% at 10% concentration and 9.091% mortality was observed at 5 per cent plant extract concentration. Extract of *Jatropha curcus* gave the mean mortality revealed that maximum mortality (25.34%) at 15% was recorded. The mean mortality was 15.67% at 10% concentration and 9.00% mortality was observed at 5% concentration of the plant extracts. Extract of *Citrus paradise* gave the mean mortality revealed that maximum mortality (14.00%) at 15% was recorded. The mean mortality was 10.00% at 10% concentration and 5.00% mortality of the plant extracts was observed at 5 percent. The given outcome showed that the interaction of exposure time and concentration was significant. From the findings, we estimated that mortality values were rising slowly, with a rise in plant extract concentration.

## Titanium Nano-composites

### 8: UV-VIS spectra analysis

The UV-Vis spectra were analysed to validate the presence of nanoparticles in the resulting solutions. UV-Vis absorption spectroscopy is an important technique for controlling the composition and consistency of aqueous solution in metal NPs. Metal NP's absorption range is susceptible to several variables like particle size, shape, and nano-particle interaction (agglomeration) with the media. Maximum absorption ( $\lambda_{max}$ ) depends on nanoparticles, thickness, and form. The UV-VIS absorption spectrum for TiO<sub>2</sub> nanoparticles between 200 and 400 nm is shown in Figure 4.3 TiO<sub>2</sub> absorption occurs at 280 nm, and its absorbance in UV – Vis spectroscopy will be 0.004. (Aryal et cetera, 2006).

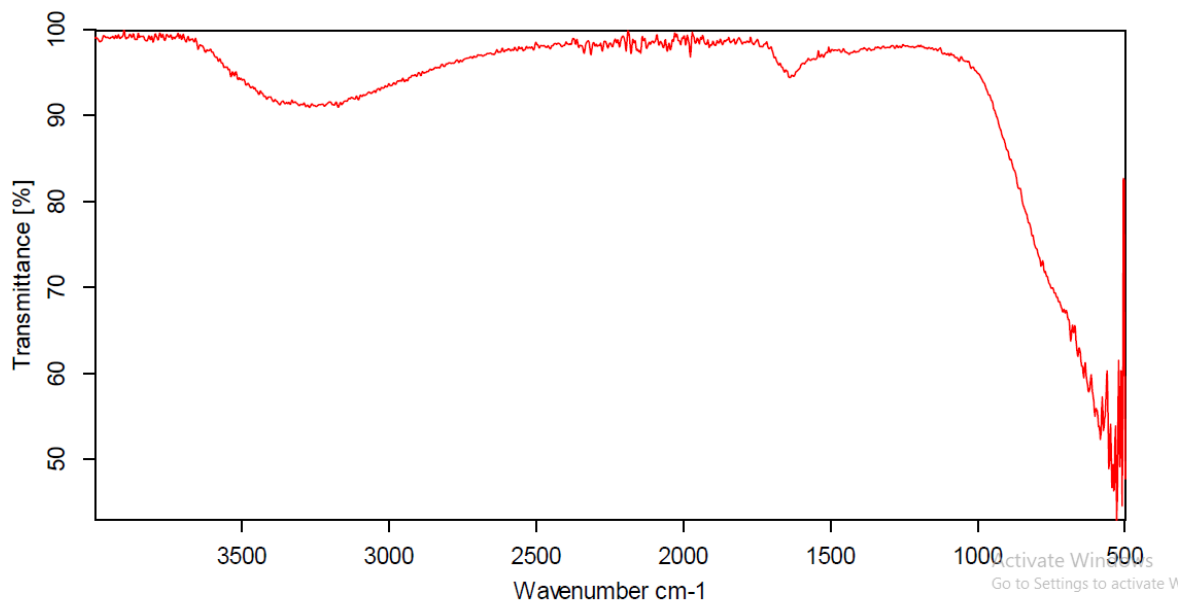


**Figure 8.1:** UV-Vis absorption spectra of titanium nanoparticle synthesized

### 9: Fourier transforms infrared spectroscopy (FTIR)

FTIR spectroscopy was used to classify various groups on *Ricinus communis* powder and to estimate their role in the formulation of nanoparticles. Figure 6 reveals the bands are at 3320 cm<sup>-1</sup>, 1590 cm<sup>-1</sup>, 1385 cm<sup>-1</sup>, 1024 cm<sup>-1</sup>. And 431-1 cm. The TiO<sub>2</sub> nanoparticles FTIR spectrum showed characteristic 1024 cm<sup>-1</sup> bands which indicate the presence of C-O stretching alcohols,

carboxylic acids, esters, and ethers. The peak at 1385 cm<sup>-1</sup> indicates C-H rock alkenes while the peak at 1590 cm<sup>-1</sup> indicates the presence of saturated hydrocarbon signature C = C. The 3320 cm<sup>-1</sup> band correlates to O-H, as do the alcohols and phenols bonded with H-. Such bonds are related to the Ricinus communis chemical composition. In addition, the structure of Ricinus communis contains such compounds as caffeic acid, quinic acid and chlorogenic acid. It also contains free-form, glycosidally bound flavonoids (quercetin, kaempferol, rutoside, luteolin, apigenin, isorhamnetin), and essential oil. The peak at 431 cm<sup>-1</sup> shows TiO<sub>2</sub> nanoparticles



are present.

**Figure 9.1** FTIR spectra of titanium dioxide nanoparticles

## Conclusion

The present data provide evidence that the method of green synthesis would also prepare modern nanotechnology. These nanoparticles were prepared using various solvents from plant extracts. The big influence on soil fertility or growth was produced by the tribolium castanium and Trigonoderma granarium pests. Specific plant extract oil has been used in recent years to regulate these effects but it won't work properly. So now we can use nanoparticles to monitor the giant reproduction disease in our country. Our experiments prove that the nanoparticles work very well, and then the oil extracts very easily. The results presented will prove this correctly. Then we can conclude that the nanoparticles recommended for farming level due to their cheap cost, availability, eco-friendly nature and strong alternative ways against pest control.

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