

# TABLE OF CONTENTS

<b>ACKNOWLEDGEMENTS</b> .....	i
<b>TABLE OF CONTENTS</b> .....	ii
<b>LIST OF ABBREVIATIONS</b> .....	vii
<b>List of tables</b> .....	viii
<b>List of figures</b> .....	x
<b>ABSTRACT</b> .....	xiv
<b>CHAPTER 1: INTRODUCTION &amp; LITERATURE REVIEW</b> .....	1
<b>1.1. INTRODUCTION:</b> .....	1
<b>1.2. THE AIM OF THE STUDY:</b> .....	1
<b>1.3. LITERATURE REVIEW:</b> .....	1
1.3.1. Factors affecting crops yield:.....	1
1.3.2. Cotton leafworm <i>Spodoptera Littoralis</i> (Boisd.) (Lepidoptera: Noctuidae):.....	2
1.3.3. Distribution of cotton leafworm:.....	3
1.3.4. The Black cutworm, <i>Agrotis ipsilon</i> (Hufnagel) (Lepidoptera: Noctuidae):.....	3
1.3.5. Distribution of the <i>Black Cutworm</i> :.....	4
1.3.6. Life cycle of both cotton leafworm and cutworm:.....	6
1.3.7. Pest control strategies:.....	7
1.3.8. Chemical control:.....	7
1.3.8.1. Biopesticides:.....	8
1.3.8.2. Role of bacteria <i>Bacillus Thuringiensis</i> as a biopesticide:.....	9
1.3.8.3. Mode of action of <i>Bacillus thuringiensis</i> (Bt):.....	10
1.3.9. Role of nanotechnology:.....	13
1.3.9.1. What is nanotechnology:.....	13
1.3.9.2. Nano and nanomaterials:.....	13
1.3.9.3. Natural nanomaterials: .....	14
1.3.9.4. Formulations and types of nanomaterials: .....	14
1.3.9.5. Applications of nanotechnology: .....	15

1.3.9.6. Nanopesticides: .....	16
1.3.9.7. Preparation methods of nanomaterials: .....	17
1.3.9.8. TiO <sub>2</sub> nanoparticles and their role in agricultural field:.....	18
<b>1.4. Our work:</b> .....	20
<b>Chapter 2: Experimental Section.</b> .....	21
<b>2.1. Materials and methods:</b> .....	21
2.1.1. Insect rearing:.....	21
2.1.2. Materials:.....	21
2.1.3. Synthesis of H-Titanate nanotubes (TNT) and H-Titanate nanosheets (TNS):.....	22
2.1.4. Synthesis of Bacillus-Titanate nanocomposites at 0.5: 1 gm percent, respectively:.....	22
2.1.5. Synthesis of Bacillus-Titanate nanocomposites at 2:1 gm percent, respectively:.....	22
2.1.6. Synthesis of sodium titanate nanotubes (TNTs):.....	23
2.1.7. Synthesis of Bt-Na-titanate nanocomposite (Bt-TNTs):.....	23
2.1.8. Synthesis of potassium titanate nanotubes (TNT <sub>k</sub> ):.....	23
2.1.9. Synthesis of Bt- potassium titanate nanocomposites (Bt-TNT <sub>k</sub> ):.....	24
2.1.10. Synthesis of Bacillus-Titanate nanocomposites at different concentrations of nanomaterial:.....	24
2.1.11. Synthesis of Bt- TiO <sub>2</sub> nanoparticles composites (Bt-TNP) at different concentrations of nanoparticles:.....	24
<b>2.2. Characterization:</b> .....	25
<b>2.3. Bioassay:</b> .....	25
2.3.1. TiO <sub>2</sub> nanotubes (TNT), nanosheets (TNS), (Bt), (Bt-TNT) and (Bt-TNS) nanocomposites at 0.5: 1 gm percent:.....	25
2.3.2. TiO <sub>2</sub> nanotubes (TNT), nanosheets (TNS), (Bt), (Bt-TNT) and (Bt-TNS) nanocomposites at 2: 1gm percent:.....	26

2.3.3. Sodium titanate nanotubes (TNTs) and (Bt-TNTs) nanocomposite:.....	26
2.3.4. Potassium titanate nanotubes (TNTk) and (Bt-TNTk) nanocomposite:.....	27
2.3.5. TiO <sub>2</sub> nanotubes (TNT), nanosheets (TNS) and their composites with (Bt) at different nanomaterial concentrations:.....	27
2.3.6. TiO <sub>2</sub> nanoparticles (TNP) and their composite with (Bt) at different nanoparticles concentrations:.....	28
2.4. Statistical analysis:.....	28
2.5. Calculating the percent of decrease and increase in the biological parameter:.....	29
<b>Chapter 3: Results and discussion.....</b>	<b>30</b>
<b>3.1. Effect of different samples on Cotton cutworm <i>Agrotis ipsilon</i> (Hufnagel) (<i>Lepidoptera: Noctuidae</i>):.....</b>	<b>30</b>
<b>3.2. Effect of (TNT), (TNS) and their composites with (Bt) at 0.5: 1 gm bacteria to nanomaterial, respectively, on the treated 2<sup>nd</sup> and 4<sup>th</sup> instars larvae of <i>S.Littoralis</i> (<i>Boisd.</i>):.....</b>	<b>30</b>
<b>3.2.1. Physical characterization:.....</b>	<b>30</b>
<b>3.2.2. Latent effect:.....</b>	<b>37</b>
3.2.2.1. Total mortality, Pupation and adult emergence %:.....	37
3.2.2.2. Larval and pupal duration :.....	37
3.2.2.3. Pupal weight:.....	43
3.2.2.4. Adult longevity and adult sex ratio:.....	43
3.2.2.5. Adult fecundity and eggs hatchability %:.....	44
<b>3.3. Effect of (TNT), (TNS) and their composites with (Bt) at 2: 1 gm bacteria to nanomaterial, respectively, on the treated 2<sup>nd</sup> and 4<sup>th</sup> instars larvae of <i>S.Littoralis</i> (<i>Boisd.</i>):.....</b>	<b>50</b>
<b>3.3.1. Latent effect:.....</b>	<b>50</b>
3.3.1.1. Total mortality, pupation and emergence percentages:.....	50
3.3.1.2. Larval duration, pupal duration and adult longevity:.....	53
3.3.1.3. Adult longevity and adult Sex ratio:.....	56
3.3.1.4. Adult fecundity and eggs hatching %:.....	56

3.3.1.5. Malformations %:.....	61
<b>3.4. Effect of sodium titanate (TNTs), (Bt) and their composite (Bt-TNTs) on the treated 2<sup>nd</sup> and 4<sup>th</sup> instars larvae of <i>S.Littoralis</i> (Boisd.):.....</b>	<b>64</b>
<b>3.4.1. Physical characterization:.....</b>	<b>64</b>
<b>3.4.2. Latent effect:.....</b>	<b>69</b>
3.4.2.1. Total mortality, pupation and emergence: .....	69
3.4.2.2. Larval and pupal duration: .....	69
3.4.2.3. Malformations :.....	74
3.4.2.4. Adult fecundity and eggs hatching %: .....	77
3.4.2.5. Adult sex ratio and adult longevity:.....	77
<b>3.5. Effect of Potassium titanate (TNT<sub>k</sub>), (Bt) and their composite (Bt-TNT<sub>k</sub>) on the treated 2<sup>nd</sup> and 4<sup>th</sup> instars larvae of <i>S.Littoralis</i> (Boisd.):.....</b>	<b>82</b>
<b>3.5.1. Physical characterization:.....</b>	<b>82</b>
<b>3.5.2. Latent effect:.....</b>	<b>86</b>
3.5.2.1. Total mortality, pupation and emergence:.....	86
3.5.2.2. Larval and Pupal duration:.....	86
3.5.2.3. Malformations:.....	91
3.5.2.4. Adult fecundity and eggs hatching %:.....	94
3.5.2.5. Adult longevity and adult sex ratio:.....	94
<b>3.6. Effect of (TNT), (TNS), (Bt) and their nanocomposites at different concentrations of nanomaterials on the treated 2<sup>nd</sup> and 4<sup>th</sup> instars larvae of <i>S.Littoralis</i> (Boisd.):.....</b>	<b>99</b>
<b>3.6.1. Latent effect:.....</b>	<b>99</b>
3.6.1.1. Total mortality, Pupation and Emergence percentages:.....	99
3.6.1.2. Larval and Pupal duration:.....	102
3.6.1.3. Malformations:.....	106
3.6.1.4. Adult fecundity and eggs hatching %:.....	108
3.6.1.5. Adult sex ratio and adult longevity:.....	111

<b>3.7. Effect (TNP), (Bt) and their nanocomposite at different concentrations of nanoparticles on the treated 2<sup>nd</sup> and 4<sup>th</sup> instars larvae of <i>S.Littoralis</i> (Boisd.):</b>	116
<b>3.7.1. Physical characterization:</b>	116
<b>3.7.2.Latent effect:</b>	122
3.7.2.1. Total mortality %, Pupation and Emergence percentages:	122
3.7.2.2. Larval and pupal duration:	125
3.7.2.3. Malformations:	129
3.7.2.4. Adult fecundity and eggs hatching %:	131
3.7.2.5. Adult longevity and sex ratios:	131
<b>CONCLUSION</b>	138
<b>REFERENCES</b>	140
<b>APPENDICES</b>	116
APPENDIX A: THE FIRIST PUBLISHED PAPER:	118
APPENDIX B: THE SECOND PUBLISHED PAPER.	124
<b>ARABIC ABSTRACT</b>	133

## LIST OF ABBREVIATIONS:

<b>TiO<sub>2</sub></b>	<b>Titanium Dioxide</b>
<b>Bt</b>	<b>Bacillus Thuringiensis</b>
<b>TNT</b>	<b>H-Titanate Nanotubes</b>
<b>TNS</b>	<b>H-Titanate Nanosheets</b>
<b>Bt-TNT</b>	<b>Bacillus-TNT Nanocomposite</b>
<b>Bt-TNS</b>	<b>Bacillus-TNS Nanocomposite</b>
<b>TNT<sub>s</sub></b>	<b>Sodium Titanate Nanotubes</b>
<b>TNT<sub>k</sub></b>	<b>Potassium Titanate Nanotubes</b>
<b>TNP</b>	<b>TiO<sub>2</sub> Nanoparticles</b>
<b>Bt-TNP</b>	<b>Bacillus-TNP Nanocomposite</b>
<b>X1</b>	<b>1 gm/L concentration</b>
<b>X2</b>	<b>0.75 gm/L concentration</b>
<b>X3</b>	<b>0.5 gm/L concentration</b>
<b>X4</b>	<b>0.25 gm/L concentration</b>
<b>M</b>	<b>Males</b>
<b>F</b>	<b>Females</b>

## List of tables

Table.1. Effect of (TNT), (TNS), (Bt) and their composites on larval and pupal mortalities, pupation and emergence of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars compared to control.....	38
Table.2. Effect of (TNT, (TNS), (Bt) and their composites on the larval and pupal duration, and pupal weight of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars compared to control. ....	40
Table.3. Effect of (TNT), (TNS), (Bt) and their composites on the adult longevity and Sex ratios of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars compared to control. ....	48
Table.4. Effect of (TNT), (TNS), (Bt) and their composites on the adult fecundity and eggs hatching of the treated 4 <sup>th</sup> instar compared to control. ....	49
Table.5. Effect of (TNT), (TNS), (Bt), (Bt-TNT) and (Bt-TNS) on total mortality, pupation and emergence percentages of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars.....	52
Table.6. Effect of (TNT), (TNS), (Bt-TNT), (Bt-TNS) and (Bt) on larval and pupal duration, and malformation of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars.....	54
Table.7. Effect of (TNT), (TNS), (Bt), (Bt-TNT) and (Bt-TNS) on adult fecundity and sex ratio of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	57
Table.8. Effect of (TNT), (TNS), (Bt-TNT), (Bt-TNS) and (Bt) on adult fecundity and eggs hatching of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instar. ....	59
Table.9. Effect of (TNTs), (Bt) and (Bt-TNTs) on mortality % , pupation and emergence % of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	70
Table.10. Effect of (TNTs), (Bt) and (Bt-TNTs) on larval and pupal duration, and larval, pupal and adult malformation of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instar.....	71
Table.11. Effect of (TNTs), (Bt) and (Bt-TNTs) on adult fecundity and hatchability % of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars.....	78
Table.12. Effect of (TNTs), (Bt) and (Bt-TNTs) on adult fecundity and hatchability % of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars.....	79
Table.13. Effect of (Bt), (TNT <sub>k</sub> ) and (Bt-TNT <sub>k</sub> ) on total mortality, pupation % and adult emergence % of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instar.....	87

Table.14. Effect of (Bt), (TNT <sub>k</sub> ) and (Bt-TNT <sub>k</sub> ) on larval and pupal duration, and larval, pupal and adult malformation of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ...	88
Table.15. Effect of (TNT <sub>k</sub> ), (Bt) and (Bt-TNT <sub>k</sub> ) on adult fecundity and eggs hatching % of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars.....	95
Table.16. Effect of (TNT <sub>k</sub> ), (Bt) and (Bt-TNT <sub>k</sub> ) on adult longevity and sex ratio of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars.....	96
Table.17. Effect of (TNT), (TNS), (Bt-TNT), (Bt-TNS) at different concentrations and (Bt) at 0.5 gm / L on total mortality, pupation % and emergence % of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	102
Table.18. Effect of (TNT), (TNS), (Bt-TNT), (Bt-TNS) at different concentrations and (Bt) at 0.5 gm / L on larval and pupal duration, and larval, pupal and adult malformation of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	105
Table.19. Effect of (TNT), (TNS), (Bt-TNT), (Bt-TNS) at different concentrations and (Bt) at 0.5 gm / L on adult fecundity and eggs hatching % of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars.....	109
Table.20. Effect of (TNT), (TNS), (Bt-TNT), (Bt-TNS) at different concentrations and (Bt) at 0.5 gm / L on adult longevity and sex ratio of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	113
Table.21. Effect of (TNP), (Bt-TNP) and (Bt) on total mortality, pupation and emergence % of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	124
Table.22. Effect of (TNP), (Bt-TNP) and (Bt) on larval and pupal duration and larval, pupal and adult malformations of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars.....	128
Table.23. Effect of (TNP), (Bt-TNP) and (Bt) on a- larval, b-pupal and c- adult malformations % of treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	133
Table.24. Effect of (TNP), (Bt-TNP) and (Bt) on a- larval, b-pupal and c- adult malformations % of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	134



## List of figures

Fig.1. a-larva, b-adult moth, c- egg mass and d-pupa of the cotton leafworm <i>S.littoralis</i> (Boisd.).	4
Fig.2. a- egg masses, b- larva, c- pupa and d- adult moth of the black cutworm, <i>Agrotis ipsilon</i> (Hufnagel) (Lepidoptera: Noctuidae).	5
Fig. 3. The life cycle of cotton leafworm and cutworm.	6
Fig.4. a-Insect anatomy and b- <i>Bacillus thuringiensis</i> life cycle.	11
Fig.5. Top-down and bottom-up techniques used in nanostructures preparation.	17
Fig.6. XRD patterns of (TNT), (TNS), (Bt-TNT), (Bt-TNS) and (Bt).	32
Fig.7. FT-IR spectra of (TNT), (TNS), (Bt-TNT), (Bt-TNS) and (Bt).	33
Fig.8. FESEM of a- (Bt), b-(TNT), c-(TNS), d- (Bt-TNT) and e- (Bt-TNS).	35
Fig.9. The average zeta-potentials of (TNT), (TNS), (Bt) and their composites.	36
Fig.10. Effect of (TNT), (TNS), (Bt) and their composites on a- total mortality %, b- pupation % and c- adult emergence % of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars compared to control.	39
Fig.11. Effect of (TNT), (TNS), (Bt) and their composites on the larval and pupal duration of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars compared to control .	41
Fig.12. Effect of (TNT), (TNS), (Bt) and their composites on the pupal weight of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars compared to control .	42
Fig.13. Effect of (TNT), (TNS), (Bt) and their composites on the adult longevity of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars compared to control. .	45
Fig.14. Effect of (TNT), (TNS), (Bt) and their composites on the adult sex ratio of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars compared to control. .	46
Fig.15. Effect of (TNT), (TNS), (Bt) and their composites on a- adult fecundity and b-eggs hatching of the treated 4 <sup>th</sup> instar compared to control .	47

Fig.16. Effect of (TNT), (TNS), (Bt), (Bt-TNT) and (Bt-TNS) on a-total mortality, b- pupation and c- adult emergence percentages of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars.....	51
Fig.17. Effect of (TNT), (TNS), (Bt), (Bt-TNT) and (Bt-TNS) on a- larval duration and b- pupal duration of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	55
Fig.18. Effect of (TNT), (TNS), (Bt), (Bt-TNT) and (Bt-TNS) on a- larval duration and b- pupal duration of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars.....	58
Fig.19. Effect of (TNT), (TNS), (Bt), (Bt-TNT) and (Bt-TNS) on a- adult fecundity and b-eggs hatching % of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	60
Fig.20. Effect of (TNT), (TNS), (Bt), (Bt-TNT) and (Bt-TNS) on a-larval , b-adult and c-pupal malformations of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	62
Fig.21. Different malformation degrees (a) and normal adult and pupa (b)...	63
Fig.22. (FESEM) of: a-(TNTs) , b-(Bt) and c-(Bt-TNTs). ....	66
Fig.23. XRD patterns of (TNTs), (Bt) and (Bt-TNTs). ....	67
Fig.24. FTIR spectra of (TNTs), (Bt) and (Bt-TNTs).....	67
Fig.25. Zeta potentials of (TNTs), (Bt) and (Bt-TNTs).....	68
Fig.26. Effect of (TNTs), (Bt-TNTs) and (Bt) on a- total mortality, b- pupation and c- adult emergence % of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	72
Fig.27. Effect of (TNTs), (Bt-TNTs) and (Bt) on a- larval and b-pupal duration of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars.....	73
Fig.28. Effect of (TNTs), (Bt-TNTs) and (Bt) on the adult malformation of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	75
Fig.29. Different malformation degrees (a) and normal adult moth and pupa (b). ....	76
Fig.30. Effect of (TNTs), (Bt-TNTs) and (Bt) on a- adult fecundity and b - eggs hatching % of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	80
Fig.31. Effect of (TNTs), (Bt-TNTs) and (Bt) on a- adult fecundity and b-adult sex ratio of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	81

Fig.32. FESEM of a- (Bt), b- (TNT <sub>k</sub> ) and c- (Bt-TNT <sub>k</sub> ).....	83
Fig.33. Zeta potentials of (TNT <sub>k</sub> ), (Bt) and (Bt-TNT <sub>k</sub> ). .....	84
Fig.34. XRD - patterns of (TNT <sub>k</sub> ), (Bt) and (Bt-TNT <sub>k</sub> ).....	85
Fig.35. FTIR - spectra of (TNT <sub>k</sub> ), (Bt) and (Bt-TNT <sub>k</sub> ).....	85
Fig.36. Effect of (TNT <sub>k</sub> ), (Bt-TNT <sub>k</sub> ) and (Bt) on a- total mortality, b- pupation % and c- adult emergence % of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars.....	89
Fig.37. Effect of (TNT <sub>k</sub> ), (Bt-TNT <sub>k</sub> ) and (Bt) on larval and pupal duration of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	90
Fig.38. Effect of (TNT <sub>k</sub> ), (Bt-TNT <sub>k</sub> ) and (Bt) on a- pupal and b- adult malformations of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	92
Fig.39. Different malformation degrees (a) and normal adult and pupa (b)...	93
Fig.40. Effect of (TNT <sub>k</sub> ), (Bt-TNT <sub>k</sub> ) and (Bt) on a- adult fecundity and b- eggs hatching % of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	97
Fig.41. Effect of (TNT <sub>k</sub> ), (Bt-TNT <sub>k</sub> ) and (Bt) on a- adult fecundity and b- sex ratio % of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	98
Fig.42. Effect of (TNT), (TNS), (Bt-TNT), (Bt-TNS) at different concentrations and (Bt) at 0.5 gm / L on a- total mortality, b- pupation % and c- adult emergence % of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	101
Fig.43. Effect of (TNT), (TNS), (Bt-TNT), (Bt-TNS) at different concentrations and (Bt) at 0.5 gm / L on larval and pupal duration of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	104
Fig.44. Effect of (TNT), (TNS), (Bt-TNT), (Bt-TNS) at different concentrations and (Bt) at 0.5 gm / L on larval, pupal and adult malformation of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars.....	107
Fig.45. Effect of (TNT), (TNS), (Bt-TNT), (Bt-TNS) at different concentrations and (Bt) at 0.5 gm / L on adult fecundity and eggs hatching % of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars.....	110
Fig.46. Effect of (TNT), (TNS), (Bt-TNT), (Bt-TNS) at different concentrations and (Bt) at 0.5 gm / L on the adult longevity. ....	114

Fig.47. Effect of (TNP), (TNS), (Bt-TNP), (Bt-TNS) at different concentrations and (Bt) at 0.5 gm / L on adult sex ratios of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars.....	115
Fig.48. FESEM of a- (TNP), b- (Bt) and c- (Bt-TNP).....	118
Fig.49. Average zeta potentials of (TNP), (Bt) and (Bt-TNP).....	119
Fig.50. XRD patterns of (TNP), (Bt) and (Bt-TNP).....	120
Fig.51. FTIR spectra of (TNP), (Bt) and (Bt-TNP). ....	121
Fig.52. Effect of (TNP), (Bt-TNP) and (Bt) on a- total mortality, b-pupation and c- adult emergence % of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	123
Fig.53. Effect of (TNP), (Bt-TNP) and (Bt) on a- total mortality, b-pupation and c- emergence % of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	127
Fig.54. Effect of (TNP), (Bt-TNP) and (Bt) on a- larval, b-pupal and c- adult malformations % of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars. ....	130
Fig.55. Effect of (TNP), (Bt-TNP) and (Bt) on a- adult fecundity and b-eggs hatching of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars.....	135
Fig.56. Effect of (TNP), (Bt-TNP) and (Bt) on adult longevity of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars.....	136
Fig.57. Effect of (TNP), (Bt-TNP) and (Bt) on adult sex ratios of the treated 2 <sup>nd</sup> and 4 <sup>th</sup> instars.....	137

## **ABSTRACT:**

The cotton crop is one of the most important strategic crops in Egypt, but there are several types of insect pests which are very harmful to this crop. From these pests are the black cutworm, *Agrotis ipsilon* (Hufnagel) (Lepidoptera: Noctuidae) and cotton leafworm *Spodoptera littoralis* (Boisd.) (Lepidoptera: Noctuidae). Through the past decades and until now synthetic chemical pesticides such as parathion, organo-phosphorous, and synthetic pyrethroid have been used in this field to resist this pest, but the main problem comes from the extensive use of large amounts of these pesticides which cause environmental pollution. Some trials were performed to use natural alternative pesticides called biopesticides which are either bacteria, fungi or viruses. Bacteria are the most used biopesticide especially *Bacillus Thuringiensis* Subspecies *Kurstaki* (BTK), but the problem is the low activity of *Bacillus* against cotton leafworm and cutworm. Herein, for the first time the role of nanotechnology comes to make good use of some ecofriendly nanomaterials with its unique physical, chemical and electrical properties to enhance the activity of *Bacillus Thuringiensis* (Bt) in the form of nanomaterial-based biopesticide. In the present study, we will focus on the use of TiO<sub>2</sub> nanotubes (TNT), nanosheets (TNS), nanoparticles (TNP), sodium titanate (TNTs) , and potassium titanate (TNT<sub>k</sub>) alone and as nanocomposites with *Bacillus* (Bt) to study their insecticidal activity against the above mentioned pests. Also the effect of the used nanomaterials on *Bacillus* activity and their impacts on the different biological features of the two worms such as larval and pupal mortality, adult longevity, adult sex ratio, pupation, fecundity and percent of eggs hatching were studied.