

5. SUMMARY

The present work was carried out to evaluate the efficacy of three nanoparticles nano-malathion, organic nano-silica, inorganic nano-silica, and Nano-Jojoba oil were evaluated under laboratory conditions as stored product insecticide in comparison with conventional forms, by mixing with grains against two species of stored product insects the rice weevil *Sitophilus oryzae* (L.) (Curculionidae, Coleoptera); and the red flour beetle *Tribolium castaneum* (Herbst.) (Tenebrionidae, Coleoptera). Also, evaluate the effect of tested materials, residue in wheat on biochemical parameters and histological changes in different albino rat organs. Tests were performed in the stored product pests Laboratory at the Plant Protection Department, Faculty of Agriculture, Benha University.

The obtained results may be summarized as following:

1. Efficacy of the conventional malathion and it's formulated nano-malathion against *Tribolium castaneum*:

Conventional malathion: the mortality percentage was increased with increase of exposure times at all treated treatment. After 10 days from the initial treatment, mortalities were between 100, 63.6% at all tested concentrations of malathion. Reduction in the progeny ranged from 97.6 to 55.4% at various tested concentrations of malathion.

Nano malathion: the mortality percentage was increased with increase of exposure times at all treated treatment. After 7 days from the initial treatment, mortalities were between 100, 66.7% at all tested concentrations of nano malathion. Reduction in the progeny ranged from 97 to 71.5% at various tested concentrations of nano malathion.

2. Efficacy of conventional malathion and it's nano formulated malathion against *Sitophilus oryzae*:

Conventional malathion:

The mortality percentage was increased with increase of exposure times at all treated treatment. After 12 hours from the initial treatment, mortalities were between 100, 3.3% at all tested concentrations of malathion.

Nano malathion:

The mortality percentage was increased with increase of exposure times at all treated treatment. After 12 hours from the initial treatment, mortalities were between 100, 26.7% at all tested concentrations of nano malathion.

3. Efficacy of nano-silica against *Tribolium castaneum*:

Organic nano-silica:

The results concerning the toxic effects of organic nano silica on adult mortality and reduction in progeny of *Tribolium castaneum*, the mortality percentage was increased with increase of exposure times at all treated treatment. After 14 days from the initial treatment, mortalities were between 62.2, 23.3% at all tested concentrations of nano organic silica. Reduction in the progeny ranged from 92.9 to 65.01% at various tested concentrations nano organic silica.

Inorganic nano-silica:

The mortality percentage was increased with increase of exposure times at all treated treatment. After 14 days from the initial treatment, mortalities were between 100, 48.9% at all tested concentrations of nano inorganic silica. Reduction in the progeny ranged from 100 to 78.4% at various tested concentrations nano inorganic silica.

4. Efficacy of nano-silica against *Sitophilus oryzae*:

Organic nano silica:

The mortality percentage was increased with increase of exposure times at all treated treatment. After 14 days from the initial treatment, mortalities were between 85.6, 32.2% at all tested concentrations of nano organic silica. Reduction in the progeny ranged from 94.09 to 76.01% at various tested concentrations nano organic silica.

Inorganic nano silica:

The mortality percentage was increased with increase of exposure times at all treated treatment. After 14 days from the initial treatment, mortalities were between 46.7, 23.2% at all tested concentrations of nano inorganic silica. Reduction in the progeny ranged from 93.4 to 67.9% at various tested concentrations nano inorganic silica.

5. Efficacy of essential oil against *Tribolium castaneum*:

Jojoba oil:

The mortality percentage was increased with increase of exposure times at all treated treatment. After 14 days from the initial treatment, mortalities were between 48.9, 13.3% at all tested concentrations of jojoba. Reduction in the progeny ranged from 69.9 to 35% at various tested concentrations of jojoba essential oil.

Nano jojoba oil:

The mortality percentage was increased with increase of exposure times at all treated treatment. After 5 days from the initial treatment, mortalities were between 100, 7.8% at all tested concentrations of nano jojoba. Reduction in the progeny ranged from 96.9 to 34.9% at various tested concentrations of nano jojoba essential oil.

6. Efficacy of essential oil against *Sitophilus oryzae*:

Jojoba oil:

The mortality percentage was increased with increase of exposure times at all treated treatment. After 10 days from the initial treatment, mortalities were between 100, 13.3% at all tested concentrations of jojoba. Reduction in the progeny ranged from 65.6 to 25.5% at various tested concentrations of jojoba essential oil.

Nano jojoba oil:

The mortality percentage was increased with increase of exposure times at all treated treatment. After 3 days from the initial treatment, mortalities were between 100, 7.8% at all tested concentrations of nano jojoba. Reduction in the progeny ranged from 96.4 to 33.8% at various tested concentrations of nano jojoba essential oil.

7. Biosafety results:

7.1 Serum biochemical parameters:

The results of biochemical parameters of liver and kidney function upon 10 days after the exposure are showed The administration of jojoba, nano jojoba, malathion, nano malathion and nano silica caused severe hepato and reno-toxicity in the treated rats, as evidenced by the significant decrease of serum ALP levels, while was compared to control animals.as well as, on CREAT level which was significantly increase for jojoba and nano jojoba while, it was significantly decrease for nano silica.

And non-significantly for malathion and nano malathion. But on ALT was significantly increase for all treatments.

After 20 days the results significant decrease of serum ALP levels for nano jojoba and malathion. While, it was significantly increase for jojoba, nano malathion and nano silica while was compared to control animals.as well as, on CREAT level which was significantly increase for nano malathion while, it was significantly decrease for jojoba, nano jojoba and nano silica. And non-significantly for malathion. But on ALT was significantly increase for all treatments. While, after 30 days significant decreases ($P<0.05$) in ALP were recorded in jojoba, nano jojoba, malathion, nano malathion and nano silica as compared to untreated control group (3.01, 3.17, 3.32, 3.04, 3.42 and 3.72 respectively), while, on ALT and CREAT they were showed a significant increase ($P<0.05$) as compared to untreated control group (83.5, 61.5, 59.00, 61.00, 59.00 and 23.50 respectively) for AIT, and (0.45, 0.60, 0.55, 0.45, 0.61 and 0.40 respectively) for CREAT.

7.2 Results of hematological parameters:

The effects of jojoba, nano jojoba, malathion, nano malathion and nano silica on serum biochemical analysis after 10 days. There was significantly decrease in the WBC, MCV and HCT as compared to untreated control group. As well as it was significantly decrease in the HGB for jojoba, nano jojoba and malathion, but it was significantly increase for nano malathion and nano silica. And it was significantly decreased in the MCH for all treatments except for nano jojoba which was significantly increased. In the RBC and PLT it was significantly increase in all treatments except for nano jojoba in the RBC which was significantly decrease. Additionally it was significantly increase for jojoba and nano jojoba but it was significantly decrease for malathion, nano malathion and nano silica in the MCHC.

The effects of jojoba, nano jojoba, malathion, nano malathion and nano silica on serum biochemical analysis after 20 days. Data showed in WBC significantly decrease for jojoba, nano jojoba and malathion but there was significantly increase for nano malathion and nano silica as compared with control. As well as it was significantly increase in HGB, RBC, MCHC and PLT for all treatments. But it was significantly decrease in MCV and HCT for all treatments as compared with control.

The effects of jojoba, nano jojoba, malathion, nano malathion and nano silica on serum biochemical analysis after 30 days. Data showed in WBC significantly

increase for all treatments. As well as, it was significantly increased in HGB, RBC, MCHC and PLT for all treatments. But it was significantly decrease in MCV and HCT for all treatments as compared with control.

8. Histological changes in rat's organs due to the tested compounds:

8.1 Treatment of jojoba oil:

Kidneys after 10 days the kidneys of rats of this group severe shrinkage of glomerular tuft were noticed. Moreover, after 20 days the kidneys showed severe congestion and diltation of the renal blood vessels, as well as, perivascular edema. Additionally, after 30 days severe degeneration and segmentation of their glomerular tuft with thickening of their Bowman's capsule were seen in some of the affected glomeruli. As well, severe degenerative changes in the form of vaculation and desquamation of their epithelial Cell lining of the renal tubules were detected with presence of esinophilic debris in their luminae.

Liver The liver of rats of this group after 10 and 20 days showed the same result that the hepatocytes were suffering from severe degree of vacuolar and hydropic degeneration. And after 30 days there were severe congestion and diltation of the central vein and hepatic sinusoids. Moreover, few fatty vacuoles were seen in the cytoplasm of hepatocyte that squeezing nucleus to one side giving signet ring shape.

Testis after 10 days the testis of this group showed mild degeneration in their germinal epithelium. After 20 and 30 days showed the same results.

8.2 Treatment of nano jojoba oil:

Kidneys the kidneys of rats of this group showed after 10 days that the shrinkage of the glomerular tuft with severe vaculation of their epithelial cell lining was observed. Moreover, severe degenerative changes in the form of cloudy swelling that characterized by swollen cell lining with narrow star shaped lumen. With granular esinophilic cytoplasm were seen in most of renal tubules. Moreover, after 20 and 30 days there were the same results which showed severe congestion and diltation of the renal blood vessels.

Liver after 10 days the liver showed mild diltation of central vains and hepatic sinusoids. Moreover, after 20 days the hepatocytes were suffering from severe degree

of hydropic degeneration. Additionally after 30 days the portal areas showed severe diltation of the portal blood vessels with mild hyperplasia with periductal fibrosis around bile duct.

Testis the results after 10 days were similar to results after 20 and 30 days and it showed that the testis of rats of this group showed mild thickening of tunica albuginea with mild inter- tubular edema. Moreover, severe degree of degeneration and vaculation of germinal epithelium of some seminiferous tubules were detected.

8.3 Treatment of malathion:

Kidneys after 10days the kidneys microscopically showed severe congestion and diltation of the renal blood vessels and inter tubular blood capillaries. Additionally, after 20days severe degenerative changes which manifested by severe desquamation and vaculation of the epithelial cell lining of renal tubules with presence of esinophilic debris in their luminae with pyknotic nuclei was seen. Moreover, after 30 days proliferation of the glomerular tuft with the presence of inter tubular hemorrhage was detected.

Liver The microscopical examination of the liver of rats in this group after 10 days showed that the portal area showed severe congestion of the portal blood vessels with mild hyperplasia and periductal fibrosis of bile duct these changes were similar to changes which was found after 20 days. Moreover, after 30 days revealed congestion and diltation of central vain and hepatic blood sinusoids.

Testis After 10 days the testis of this group showed mild formations of sperm gaint cell in the lumen of the affected seminiferous tubules were also seen. After 20 days mild thickening of tunica albuginea with mild congestion of the capsular blood vessels. Moreover, mild interstitial edema was also detected. Additionally, mild degenerative changes in the germinal epithelium that lining the seminiferous tubules were also seen. And after 30 days degeneration of the germinal epithelium with desquamation of most spermatogenic cells in the lumen of most of the seminiferous tubules were observed.

8.4 Treatment of nano malathion:

Kidneys After 10 days the kidneys of rats in this group showed severe degeneration and proliferation of glomerular tuft with inter tubular hemorrhage. Moreover, vesiculations of the glomerular tuft with mild thickening of Bowman's capsule as well as periglomerular hemorrhage were also detected. And after 20 days the renal tubules were suffered from severe degenerative changes in the lining epithelium with complete desquamation of lining epithelium with pyknotic nuclei nearly in all renal tubules. Additionally, after 30 days the results showed severe degeneration and proliferation of glomerular tuft with inter tubular hemorrhage.

Liver After 10 days the liver of rats of this group showed severe dilation of the central veins and hepatic sinusoids. Moreover, focal areas of hemorrhages in the hepatic parenchyma with focal mononuclear cellular aggregations were seen. The affected hepatocytes showed mild fatty changes in which the intra - cytoplasmic clear fat vacuoles pushing nucleus to one side give characteristic signet ring appearance. Additionally, severe degree of vacuolar and hydropic degeneration was also observed in the hepatocyte. After 20 days the portal areas showed severe congestion and dilation of the portal veins, mild degree of hyperplasia of the biliary epithelium with mild periductal fibrosis was observed these were similar to changes which were seen after 30 days.

Testis After 10 days the testis showed severe degree of degeneration of germinal epithelium, lining the seminiferous tubules as primary spermatocyte, secondary spermatocyte, spermatid and spermatozoa. Moreover, after 20 days complete necrosis and hyalinosis of germinal epithelial cell lining of some seminiferous tubules was also demonstrated. Additionally, after 30 days Apoptotic changes were seen in most of seminiferous tubules.

8.5 Treatment of nano silica:

Kidneys of rat of this group after 10 days showed that most of the renal tubules were also suffered from severe necrotic changes represented by pyknotic nuclei and cytoplasmolysis. Moreover, after 20 days there were severe congestion and dilation of the renal blood vessels, and inter tubular blood capillaries. Additionally, after 30 days focal area of necrosis represented by complete degeneration of the renal tubules infiltrated with mono nuclear leukocytic infiltration.

Liver after 10 days the liver showed that the portal areas showed periductal fibrosis as well as mono nuclear leukocytic infiltration. Additionally, after 20 days

the liver showed congestion and diltation of central veins and portal blood vessels. Moreover, focal areas of necrosis in the hepatic parenchyma were also detected and these results were similar to results after 30 days.

Testis The results after 10 days were similar to results after 20 and 30 days and it showed that the testis of rats of this group showed thickening of tunica albuginea with mild congestion of capsular blood vessels. Moreover, severe degree of degeneration of germinal epithelium that lining the seminiferous tubules was also seen.

