SYNERGISM OF *Azadirachta indica* A. Juss. AND *Piper sarmentosum* Roxb. EMULSION FORMULATION AGAINST *Nilaparvata lugens* (Stål.)

By

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Nilaparvata lugens, brown planthopper is an important rice pest that destroys rice by feeding on the plant sap and causing a condition known as 'hopper burn', where the plants are completely wilted, yellow and dried. The constant use of chemical substances had built the resistance of pests toward chemical insecticide. As an alternative, a more environmentally-friendly approach such as the use of biopesticide should be implemented. Biopesticide formulation which combined more than one active ingredient to produce joint toxicity effect has gaining attention lately for its effectiveness. Thus, this study aims to determine the synergism effect of *Piper sarmentosum* and *Azadirachta indica* against *N. lugens*. *Piper sarmentosum* is a common herb used in cooking while *A. indica* is known for its medicinal properties. Bioassay of the extracts against *N. lugens* nymphs showed a synergistic effect from treatments T6, T7 and T9, with mixture ratio of *P. sarmentosum* to *A. indica* at 6:4, 7:3 and 9:1. T9 caused highest nymph mortality after 72 hours treatment. Emulsion formulations were developed by mixing 5 % extracts combination from T6, T7 and T9 with 14.25 % carrier oil (Edenor), 76 % surfactant (Tween 80 and Termul 1284), and 4.75 % water. Six formulations coded as F1, F2, F3, F4, F5 and F6 had been developed. Characterization, including stability, particle size, zeta potential, PDI value, pH, and viscosity analysis, resulted in ideal nano emulsion size ranged from 202.5 to 635.9 nm. Stability and thermostability tests that were performed and successfully produced stable and homogenous nano emulsion. F4 can be selected as the best formulation because it has the best zeta potential (-65.0 mV) and viscosity value (67.2 mPa/s), the size is in nano emulsion size range (415.3 nm), remained homogenous for centrifugation and stability test, PDI value indicated it is in ideal monodisperse phase (0.418), low surface tension (35.2 mN/m) and low pH (4.0). The formulations were then used in nymph mortality bioassay test on third instar *N. lugens* nymphs to evaluate the formulation's efficacy against *N. lugens*. The lethal concentration, LC_{50} and sub lethal, LC_{10} and LC_{25} were determined from bioassay. LC_{50} can control at least half of the treated population, while LC_{10} and LC_{25} can control 10% and 25% of

population. Highest mortality can be seen on F2 (0.043 mg/l) and the lowest was F5 (0.117 mg/l). The value of lethal and sub lethal concentration were used in a test against female *N. lugens* to determine the effect of formulations on fecundity of *N. lugens*. There are significant differences on the number of eggs produced after treatments. F4 at LC_{50} was found to cause most effective effect as it produced the least number of eggs produced, 114 eggs. In conclusion, stable formulation can be developed following the current results and findings. These will lead to the synergism efficacy of *A. indica* and *P. sarmentosum* against *N. lugens* through emulsion formulations as joint toxicity could have a better potency in controlling pests, compared to individual active ingredient.

Abstrak tesis yang dikemukakan kepada senat Universiti Putra Malaysia sebagai memenuhi keperluan untuk Ijazah Master Sains

SINERGISMA FORMULASI EMULSI *Azadirachta indica* A. Juss. DAN *Piper sarmentosum* Roxb. TERHADAP *Nilaparvata lugens* (Stål)

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Nilaparvata lugens, bena perang merupakan perosak padi penting yang memusnahkan padi dengan memakan getah tanaman dan menyebabkan keadaan dikenali sebagai 'hopper burn', di mana tumbuhan menjadi sangat layu, menguning dan kering. Penggunaan bahan kimia yang berterusan telah menyebabkan kerintangan perosak terhadap racun serangga kimia. Sebagai alternatif, pendekatan yang lebih mesra alam seperti penggunaan biopestisid perlu dilaksanakan. Formulasi biopestisid yang menggabungkan lebih daripada satu bahan aktif untuk menghasilkan kesan gabungan toksik telah mendapat perhatian ketika ini kerana keberkesanannya. Justeru, kajian ini bertujuan untuk menentukan kesan sinergisma *Piper sarmentosum* dan *Azadirachta indica* terhadap *N. lugens. Piper sarmentosum* ialah herba yang biasa digunakan dalam masakan manakala *A. indica* terkenal dengan khasiat perubatannya. Bioassai ekstrak terhadap nimfa *N. lugens* menunjukkan kesan sinergistik daripada rawatan T6, T7 dan T9, dengan nisbah campuran *P. sarmentosum* kepada *A. indica* pada 6:4, 7:3 dan 9:1. T9 menyebabkan kematian nimfa tertinggi selepas 72 jam rawatan. Formulasi emulsi telah dihasilkan dengan mencampurkan 5% ekstrak daripada kombinasi ekstrak T6, T7 dan T9 dengan 14.25% minyak, 76% surfaktan dan 4.75% air. Enam formulasi berjaya dihasilkan dengan kod F1, F2, F3, F4, F5 dan F6. Pencirian termasuk kestabilan, saiz zarah, potensi zeta, nilai PDI, pH dan analisis kelikatan telah dilakukan dan menghasilkan saiz emulsi nano yang ideal dengan julat 202.5 nm hingga 635.9 nm. Ujian kestabilan dan kestabilan suhu dilakukan dan berjaya menghasilkan emulsi nano yang stabil dan homogen. F4 menunjukan formulasi terbaik kerana mempunyai nilai potensi zeta terbaik (-65.0 mV) dan nilai kelikatan (67.2 mPa/s), berada dalam julat saiz emulsi nano (415.3 nm), kekal stabil selepas ujian kestabilan dan kestabilan suhu, nilai PDI menunjukkan ia berada dalam fasa penyebaran mono (0.418), ketegangan permukaan yang rendah (35.2 mN/m) dan pH yang rendah (4.0). Bioassai formulasi terhadap nimfa N. lugens instar ketiga telah dijalankan untuk menilai keberkesanan formulasi terhadap *N. lugens*. Kepekatan maut, LC_{50} dan

kepekatan separa maut, LC_{10} dan LC_{25} ditentukan daripada bioassai. LC_{50} boleh mengawal sekurang-kurangnya separuh daripada populasi yang dirawat, manakala LC_{10} dan LC_{25} boleh mengawal 10% dan 25% daripada populasi. Kematian tertinggi boleh dilihat pada F2 (0.043 mg/l) dan paling rendah pada F5 (0.117 mg/l). Nilai konsentrasi maut dan separa maut digunakan untuk menilai kesuburan *N. lugens* betina. Terdapat perbezaan ketara terhadap bilangan telur yang dihasilkan selepas rawatan. F4 pada LC_{50} menunjukkan kesan paling efektif kerana menyebabkan penghasilan telur yang paling sedikit, 114 telur. Kesimpulannya, formulasi yang stabil boleh dihasilkan berdasarkan keputusan dan penemuan kajian. Ini membuktikan keberkesanan sinergi *A. indica* dan *P. sarmentosum* terhadap *N. lugens* melalui formulasi emulsi kerana ketoksikan gabungan mempunyai potensi yang lebih baik dalam mengawal perosak, berbanding dengan bahan aktif individu.

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This thesis was submitted to the Senate of Universiti Putra Malaysia and has been accepted as fulfilment of the requirement for the degree of master. The members of the Supervisory Committee were as follows:

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LIST OF ABBREVIATIONS

ANOVA	Analysis of variance
BPH	Brown Plant Hopper
CI	Combination Interaction
CRD	Complete Randomized Design
DLS	Dynamic light scattering
FAO	Food and Agricultural Organization
HLB	Hydrophilic-Lipophilic Balance
IRAC	Insecticide Resistance Action Committee
LC50	Lethal concentration
LD50	Lethal dose
MoA	Mode of Action
mV	millivolt
nm	Nanometre
PDI	Polydispersity index
rpm	Rotation per minute
SE	Standard Error
USD	United State Dollar

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CHAPTER 1

INTRODUCTION

1.1 Introduction

Brown planthopper (BPH), *Nilaparvata lugens* is an important rice pest in East and Southeast Asia especially in temperate and tropical regions (Hu et. al., 2014). *Nilaparvata lugens* caused massive damage on rice crop by directly feeding on the plant phloem which leads to browning, drying and wilting of the plant (Tang et al., 2010). According to Catindig et al. (2010), infestation of *N. lugens* in Malaysia is not as serious compared to other Asian countries because the highest area damaged by *N. lugens* in 2002 was only 7,259 hectares and the lowest was 3,708 hectares in 1999.

The most used control method of *N. lugens* is by using chemical control but over the last decades *N. lugens* was found to build resistance against chemical pesticide (Datta et al., 2021). The high level of resistance to imidacloprid resulted in chemical control failure and great yield loss in 2005 (Wang et. al., 2008). The constant and unsystematic use of one insecticide has resulted in the quick development of insecticide resistance and exhaustion of most insecticide alternative in many rice-growing country (Chen et al., 2013). Therefore, it is important to explore other alternatives to control *N. lugens*.

Biopesticides are natural substances that biologically affect insect pests but cause no harm to natural ecosystem (Sporleder and Lacey, 2013; Meshram et al., 2022). One of biopesticide class is biochemical biopesticides, the naturally occurring compounds that possess active ingredients which control pest and safe toward target pest, environment and humans. Examples are essential oil, insect growth regulators, natural minerals and plant extracts (Fenibo et al., 2021).

Piper sarmentosum, also called wild betel or in Malay name known as ‘kaduk’, is plant from order Piperales and family Piperaceae (Maizatul and Nor Farahiyah, 2018). It is a perennial herb that have a creeping rhizome and striped stem that grow up to 40cm. The heart-shaped and alternate leaves are light to dark green in colour with a waxy surface (Sharifah Farhanah et al., 2016). It can be found in tropical areas of Southeast Asia, Northeast India and South China. A study by Hematpoor et al. (2017) had suggested insecticidal activity of *P. sarmentosum* extracts compound, trans-asarone, against *Sitophilus oryzae, Rhyzopertha dominica* and *Plodia interpunctella*, as it showed consistent toxicity towards these insects throughout the 60 days of observation.

Azadirachta indica also known as neem is an evergreen plant from order Sapindales and family Meliaceae, the most common medicinal plants that has got worldwide attention because of its medicinal and insecticidal properties (Sonal and Pankaj, 2014). *Azadirachta indica* usually grows up to 12 to 15 metres with tall and straight trunk and long spreading branches forming a broad round crown; it has rough dark brown bark with wide longitudinal fissures separated by flat ridges. Its leaves are compound that alternates with one another. The leaves are pinnate with short petioles, each comprising 5 to 15 leaflets and have many flowered panicles, usually in the leaf axils (Hashmat et al., 2012). The flower are protandrous, white and have a pleasant fragrance. *Azadirachta indica* exert an insecticidal effect on *Crocidolomia binotalis* larvae through antifeedant behaviour and suppression of growth (Teck et al., 2003). According to Abdullah et al. (2015), neem seeds extract can reduce the number of green leafhoppers, brown planthopper, yellow stem borer and rice-ear bug on rice plant.

There are many plant species that are known for its toxic properties in managing pest and have the potential to be developed into biopesticide products (Stevenson et al., 2017 in Tembo et al., 2018). Thus, this research was carried out to determine whether crude extract emulsion formulation derived from *A. indica* and *P. sarmentosum* have the potency to cause mortality and affect the fecundity of *N. lugens.*

1.2 Objectives

The objectives of this research are:

- To determine the synergistic effect of a mixture of *A. indica* and *P. sarmentosum* extract against *N*. *lugens*.
- To prepare emulsion formulation of *A. indica* and *P. sarmentosum* combination extract and their physical characterization
- To evaluate the insecticidal effect of the *A. indica* and *P. sarmentosum* emulsion formulations on fecundity of *N*. *lugens.*

REFERENCES

Abdelrahim, S., Elamin., & Futuwi, A.I. (2013). Insecticidal effects of neem (*Azadirachta indica* A. Juss) oils obtained from neem berries stored at different periods. *The Experiment, 6*(13), 330-337.

Abdullah, T., Gassa, A. Ngatimin, S.N.A., Agus, N. & Fattah, A. (2015). Impact of different time planting in soybeans and neem seed extract application to insect population on rice field. *Int. J. Sci. Res. Technology*, *4*(62).

Abd Jalil, M. A., Shuid, A. N., & Muhammad, N. (2012). Role of medicinal plants and natural products on osteoporotic fracture healing. Evidence-based complementary and alternative medicine *Evidence-based Complementary and Alternative Medicine*, *2012*, 714512. https://doi.org/10.1155/2012/714512

Acton, Q.A. (2011). *Nitrogen oxides: Advances in research and application*. Scholarly Editions.

Agha, M.A, Muhammad, R., Omar, D., Grozescu, I.V., Abdul Majid, Dayang Laila & Manjeri, G. (2015(. Mating behaviour of brown planthopper *Nilaparvata lugens* Stàl (Homoptera: Delphacidea) under certain biological and environmental factors. *Pakistan journal of zoology. 48.*

Agriculture. (2019). Bayer's dual trait arize 8433 hybrid rice combats insect pests as well as diseases. *23*(5).

Ahmed, S., Zafar, M. I. , Hussain, A., Riaz, M. A. , & Shahid, M. (2011). Evaluation of plant extracts on mortality and tunneling activities of subterranean termites in Pakistan. In (Ed.), *Pesticides in the Modern World - Pests control and pesticides exposure and toxicity assessment*. IntechOpen. https://doi.org/10.5772/18999

Akbari, B., Tavandashti, M.P. & Zandrahimi, M. (2011). Particle size characterization of nanoparticles – A practial approach. *Iranian Journal of Materials Science & Engineering*, *8*(2).

Alzohairy M. A. (2016). Therapeutics role of *Azadirachta indica* (Neem) and their active constituents in diseases prevention and treatment. *Evidence-based complementary and alternative medicine,* 7382506. https://doi.org/10.1155/2016/7382506

Al-Badran, A.A., Fujiwara, M., Gatlin, D.M., Mora, M.A. (2018). Lethal and sub-lethal effects of the insecticide fipronil on juvenile brown shrimp *Farfantepenaeus aztecus*. *Science Reports, 8***,** 10769. https://doi.org/10.1038/s41598-018-29104-3

Al-Rajhy, D.H., Alahmed, A.M., Hussein, H.I. & Kheir, S.M. (2003). Acaricidal effects of cardiac glycosides, azadirachtin and neem oil against the camel tick, *Hyalomma dromedarii* (Acari: Ixodidae). *Pest Management Science, 59*(11), 1250-1254. https://doi.org/10.1002/ps.748. PMID: 14620053.

Amita, P. (2007). *Production of Insecticides*. https://doi.org/10.1201/b10756-6.

Andi Trisyono, Y. & Valentina, E.F.A, Teguh Rahayu & Edhi Martono. (2017). Effects of etofenprox applied at the sublethal concentration on the fecundity of rice brown planthopper, *Nilaparvata lugens*. *Journal of Asia-Pacific* Entomology, *20*(2), 547-551. https://doi.org/10.1016/j.aspen.2017.03.013.

Anisa, A. & Nour, A.H. (2010). Effect of viscosity and droplet diameter on water-in-oil (W/O) emulsions: An experimental study. *World Academy of Science, Engineering and Technology, 38*, 691-694.

Anshuman, S. (2018). *Introduction to plastics engineering*. William Andrew Publishing, 2, 17-48. ISBN 9780323395007. https://doi.org/10.1016/B978-0-323-39500-7.00002-2.

Asib, N., Omar, Z. & Mohamed, R. (2015). Preparation, characterization and toxicity of nano-emulsion formulations of rotenone extract of *Derris elliptica*. *Journal of Chemical, Biological and Physical Sciences*, *5*(4); 3989-3997.

Atangwho, I.J, Ebong, P.E., Eyong, E.U., Asmawi, M.Z. & Ahmad, M. (2012). Synergistic antidiabetic activity of *Vernonia amygdalina* and *Azadirachta indica*: Biochemical effects and possible mechanism. *Journal of Ethnopharmacology,1 41*(3): 878-887. https://doi.org/10.1016/j.jep.2012.03.041.

Atefeh, S., Farhana, Y., Mohammad Fauzi Fahmi, K., Zarizal, S., Mohd Nasir, M.D., Mohd Khairi, H. Muhammad Nazrul, S., Corazon, A.F., Lay, K.T., Salleh, M.Z. & Zainul, A.Z. (2013). A. review on the ethnomedicinal, phytochemical and pharmacological properties of Piper sarmentosum: Scientific justification of its traditional use. *Tang Humanitas Medicine, 3* (3): 1-32.

Atiax, E, Farediah, A., Hasnah, M.S & Dayar, A. (2010). Antibacterial activity and cytotoxicity screening of Sumatran kaduk (*Piper sarmentosum* Roxb.). *Iranian Journal of Pharmacology and Therapeutics, 10*(1), 1-5.

Australian Pesticides and Veterinary Medicines Authority (APVMA). (2020). *Generation of storage stability data for agricultural chemical products*. https://apvma.gov.au/node/1042

Azmy, R., El Gohary, E.G., Gohary, E., Mahmoud, D., Salem, D. Abdou, M. & Salama, M. (2019). Assessment of larvicidal activity of nanoemulsion from *Citrus sinensis* essential oil on *Culex pipiens* L. (Diptera: Culicidae). *Egyptian Journal of Aquatic Biology and Fisheries, 23*, 61-67. 10.21608/ejabf.2019.35100.

Baboota, S., Shakeel, F., Ahuja, A., Ali, J. & Shafiq, S. (2007).Design, development and evaluation of novel nanoemulsion formulations for transdermal potential of celecoxib. *Acta Pharmaceutica*, *57*(3), 315-332. https://doi.org/10.2478/v10007-007-0025-5

Bao, Y., & Zhang, C. (2019). Recent advances in molecular biology research of a rice pest, the brown planthopper. *Journal of Integrative Agriculture, 18*(4), 716-728. https://doi.org/10.1016/S2095-3119(17)61888-4.

Bastian, E.R. (2017). Chapter 20 - Surface Tension. In Bastian E. R (Eds). Micro *and nano technologies. microfluidics: modelling, mechanics and mathematics* (pp. 421-444). Elsevier. https://doi.org/10.1016/B978-1-4557-3141-1.50020-4.

Begum, M.A., Jahan, M., Bari, M.N., Hossain M.M., & Afsana, N. (2002). Potentiality of *Micrapis discolor* (F.) as a biocontrol agent of *Nilaparvata lugens* (Stal). *Journal of Biological Sciences, 2*(9), 630-632.

Berger, D., Walters, R. & Gotthard, K. (2008). What limits insect fecundity? Body size- and temperature-dependent egg maturation and oviposition in a butterfly. *Functional Ecology*, *22*, 523 - 529. https://doi.org/10.1111/j.1365-2435.2008.01392.x.

Bhowmik, D., Chiranjib, Yadav, J., Tripathi, K.K., & Kumar, K.P. (2010). Herbal remedies of *Azadirachta indica* and its medicinal application. *Journal of chemical and pharmaceutical research, 2*, 62-72.

Bhuiyan, M.H.U., Saidur, R., Amalina, M.A., Mostafizur, R.M. & Islam, A.K.M.S. (2015). Effect of nanoparticles concentration and their sizes on surface tension of nanofluids. *Procedia Engineering, 105*, 431-437. https://doi.org/10.1016/j.proeng.2015.05.030.

Binks, B.P., Murakami, R., Armes, S.P. & Fujii, S. (2006). Effects of pH and salt concentration on oil-in-water emulsions stabilized solely by nanocomposite microgel particles. *Langmuir*, *22*(5), 2050-2057. https://doi.org/10.1021/la053017+. PMID: 16489788.

Blesson, J., Saji, C.V., Nivya R.M., & Kumar, R. (2015). Synergistic antibacterial activity of natural plant extracts and antibiotics against methicilin resistant *Staphylococcus aureus*. *World Journal of Pharmacy and Pharmaceutical Sciences, 4*(3), 741-763.

Bradshaw, C.J.A. & McMahon, C.R. (2008). Fecundity. In Sven, E.J & Brian, D.F (Eds). *Encyclopedia of Ecology*. Academic Press (pp. 1535-1543). https://doi.org/10.1016/B978-008045405-4.00645-5.

Centre for Agriculture and Bioscience International. (2022). *Invasive Species Compendium.* CAB International. www.cabi.org/isc. Retrieved February 18, 2022 from https://www.cabi.org/isc/datasheet/8112

Catindig, J.L.A, Arida, G.S., Baehaki, S.E., Bentur, J.S., Cuong, L.Q., Norowi, M., Rattanakarn, W., Sriratanasak, W., Xia, J. & Lu, Z. (2009). Situation of planthoppers in Asia. In Heong, K.L. & Hardy, B. (Eds). *Planthoppers: new threats to the sustainability of intensive rice production systems in Asia.* International Rice Research Institute.

Chen, R.H., Phuoc, T.X. & Donald, M. (2011). Surface tension of evaporating nanofluid droplets. *International Journal of Heat and Mass Transfer,*

54(11–12), 2459-2466. https://doi.org/10.1016/j.ijheatmasstransfer.2011.02.016.

Cheng, Y., Li, Y., Li, W., Song, Y., Zeng, R., & Lu, K. (2020). Effect of hepatocyte nuclear factor 4 on the fecundity of *Nilaparvata lugens*: Insights from RNA interference combined with transcriptomic analysis. *Genomics, 112*(6), 4585–4594. https://doi.org/10.1016/j.ygeno.2020.08.002

Christine, Y.S.P., Son, T., Julio, E.L., Eric, C.M., Hung, A. & Ronald, C. (2000). The effects of azadirachtin on the parasitic mite, *Varroa jacobsoni* and its host honey bee (*Apis mellifera*), *Journal of Apicultural Research, 39*(3-4), 159-168. https://doi.org/10.1080/00218839.2000.11101037

Christofi, N. (2005). Bioassays | Microbial Tests. In Paul, W.A. & Townshend, C.P. *Encyclopedia of Analytical Science (Second Edition)* (pp. 265-271). Elsevier. https://doi.org/10.1016/B0-12-369397-7/00044-3.

Daaou, M. & Bendedouch, D. (2012). Water pH and surfactant addition effects on the stability of an Algerian crude oil emulsion. *Journal of Saudi Chemical Society, 16*, 333-337. 10.1016/j.jscs.2011.05.015.

Datta, J., Wei, Q., Yang, Q., Wan, P.J., He, J.C., Wang, W.X., Lai, F.X & Ali, M.P.,& Fu, Q. (2021). Current resistance status of the brown planthopper *Nilaparvata lugens* (Stål) to commonly used insecticides in China and Bangladesh. *Crop Protection, 150*, 105789. https://doi.org/10.1016/j.cropro.2021.105789.

Dey, S., Supriya, D.zainudin, Sandipan, D., Bhaskar, M., & Pathak, S.V. (2016). Lipid nanoparticles for topical application of drugs for skin diseases, In Alexandru, M.G (Eds). *Nanobiomaterials in galenic formulations and cosmetics* (pp. 327-361). William Andrew Publishing. https://doi.org/10.1016/B978-0-323-42868-2.00013-9.

De Jong, W.H., Carraway, J.W. & Geertsma, R.E. (2012). In vivo and in vitro testing for the biological safety evaluation of biomaterials and medical devices, In Jean-Pierre, B. (Eds). *Biomaterials, biocompatibility and performance of medical devices* (pp. 120-158). Woodhead Publishing. https://doi.org/10.1533/9780857096456.2.120.

Devine, G.J. & Denholm, I. (2009). Chapter 135 - Insecticide and Acaricide Resistance. In Vincent H. R. & Ring, T.C. (Eds). *Encyclopedia of Insects (Second Edition)* (pp. 505-511). Academic Press. https://doi.org/10.1016/B978-0-12-374144-8.00144-2.

Dhaliwal, G.S., & Koul, O. (2007). *Biopesticides and pest management: Conventional and biotechnological approach*. Kalyani Publishers. New Delhi.

Dhanani, T., Shah, S., Gajbhiye, N.A. & Kumar, S. (2017). Effect of extraction methods on yield, phytochemical constituents and antioxidant activity of *Withania somnifera*. *Arabian Journal of Chemistry, 10*(1),dzolkif S1193-S1199. https://doi.org/10.1016/j.arabjc.2013.02.015.

Eduardo, B.O. & Alvaro, V. (2010). Chronic toxicity bioassay with populations of the crustacean *Artemia salina* exposed to the organophosphate diazinon. *Biological research, 43*, 357-362. https://doi.org/10.4067/S0716-97602010000300013.

Englberger, K. (2009). *Invasive weeds of Pohnpei: A guide for identification and public awareness.* Conservation Society of Pohnpei (CSP). pp29.

Essiedu, J.A., Adepoju, F.O. & Ivantsova, M.N. (2020). Benefits and limitations in using biopesticides: A review. *AIP Conference Proceedings, 2313*(1). https://doi.org/10.1063/5.0032223

Fenibo, E. O., Ijoma, G. N., & Matambo, T. (2021). Biopesticides in sustainable agriculture: A critical sustainable development driver governed by green chemistry principles. *Frontiers in Sustainable Food Systems, 5,* 141. https://doi.org/10.3389/fsufs.2021.619058

Ferreira, R.R., da Silva, L., Rodrigues, T., Lopes, G., Carneiro, E. & Pavan, B. (2018). Sublethal doses of insecticides affect the fecundity and fertility of the *Chrysodeixis includens*. *American Journal of Plant Sciences*, *9*, 483-494. https://doi.org/10.4236/ajps.2018.93036.

Fishel, F.M. & Ferrell, J.A. (2019). Water pH and the effectiveness of pesticides. *University of Florida, Institute of Food and Agricultural Sciences.*

Food and Agriculture Organization of the United Nations & World Health Organization. (2022). *Manual on the development and use of FAO and WHO specifications for chemical pesticides*: Second edition. Food & Agriculture Organization.

Gad, S.C. (2014). LD_{50}/LC_{50} (Lethal Dosage 50/Lethal Concentration 50). In Philip Wexler. *Encyclopedia of Toxicology (Third Edition)* (pp. 58-60). Academic Press. https://doi.org/10.1016/B978-0-12-386454-3.00867-8.

Gang, F., Min, C., Huo-Chun, Y., Zheng-Ke, Z., Hong, L., Li-Liang, C., Xiao-Ling, C., Chao, Y. & Jing, Z. (2019). Herbicidal activities of compounds isolated from the medicinal plant *Piper sarmentosum*. *Industrial Crops and Products, 132*, 41-47. https://doi.org/10.1016/j.indcrop.2019.02.020.

Gasparelo, A.P., Grupo, B. & São José, D.P. (2014). Zeta potential and particle size to predict emulsion stability. *Cosmetics and toiletries.*

Ghaderpoori, M., Jafari, A., Nazari, E., Rashidipour, M., Nazari, A., Chehelcheraghi, F., Kamarehie, B., & Rezaee, R. (2020). Preparation and characterization of loaded paraquat- polymeric chitosan/xantan/tripolyphosphate nanocapsules and evaluation for controlled release. *Journal of environmental health science & engineering, 18*(2), 1057–1066. https://doi.org/10.1007/s40201-020-00527-3

Ghosal, A. (2018). *Mode of Action of Insecticides*. AkiNik Publications.

Gisi, U. (1996). Synergistic interaction of fungicides in mixtures. *Phytopathology, 86*(11), 1273-1279.

Gokce, A., Whalon, M.E., Cam, H., Yanar, Y., Demirtas, I. & Goren, N. (2006). Plant extract contact toxicities to various developmental stages of Colorado potato beetles (Coleoptera: Chrysomelidae). *Annals of Applied Biology, 149*(2), 197-202.

Goodarzi, F. & Zendehboudi, S. (2019). A comprehensive review on emulsions and emulsion stability in chemical and energy industries. *The Canadian Journal of Chemical Engineering, 97,* 281-309. https://doi.org/10.1002/cjce.23336

Groves, M.J. & Freshwater, D.C. (1968). Particle-size analysis of emulsion systems. *Journal of Pharmaceutical Sciences*, *57*(8). https://doi.org/10.1002/jps.2600570802.

Gupta, A., Eral, B.,Hatton, T. & Doyle, P. (2016). Nanoemulsions: formation, properties and applications. *Soft Matter. 12*, 2826-2841. https://doi.org/10.1039/C5SM02958A.

Hamid, N. Junaid, M. & Pei, D.S. (2021). Combined toxicity of endocrine-disrupting chemicals: A review. *Ecotoxicology and Environmental Safety, 215*, 112136. https://doi.org/10.1016/j.ecoenv.2021.112136.

Hashmat, I, Hussain, A. & Shaikh, M. (2012). Neem (*Azadirachta indica* A. Juss)- A nature's drugstore: An overview. *International Research Journal of Biological Sciences, 1*(6), 76-79.

Hazreen, O., Saiful Irwan, Z., Mohd Faizulhelmi, F. Dzolkhifli, O. & Norhayu, A. (2016). Preparation and physicochemical evaluation of emulsified virgin coconut oil (VCO) *Carica* papaya extract concoction using Tween80. *AIP Conference Proceeding*. *1784*, 030035-1–030035-7. doi: 10.1063/1.4966773

Helen, T. & Grahame, J, n.d. *Planthopper*. Rice Knowledge Bank. IRRI. (http://www.knowledgebank.irri.org/training/fact-sheets/pest-management/insects/item/planthopper).

Hematpoor, A., Yee Liew, Mohd Sofian Azirun & Khalijah, A. (2017). Insecticidal activity and the mechanism of action of three phenylpropanoids isolated from the roots of *Piper sarmentosum* Roxb. *Science Reports, 7*, 12576 https://doi.org/10.1038/s41598-017-12898-z

Hematpoor, A., Azirun, M.S., & Khalijah, A. (2013). Isolation and identification of active compounds from *Piper sarmentosum* against several storage pests. *The Open Conference Proceedings Journal, 4*(1), 37. http://dx.doi.org/10.2174/2210289201304010037

Hussain, K., Ismail, Z., Sadikun, A. & Ibrahim, P. (2009). Antioxidant, anti-tb activities, phenolic and amide contents of standardised extracts of *Piper sarmentosum* Roxb. *Natural Product Research, 23*(3), 238-249.

Hu, G., Lu, F., Zhai, B.P., Lu, M.H., Liu, W.C., Zhu, F., Wu, X.W., Chen, G.H. & Zhang, X.X. (2014). Outbreaks of the brown planthopper *Nilaparvata lugens* (Stål) in the Yangtze river delta: Immigration or local reproduction? *PLOS ONE*, *9*(2), e88973. https://doi.org/10.1371/journal.pone.0088973

Hu, M., Xie, F., Zhang, S., Qi, B. and Li, Y. (2021). Effect of nanoemulsion particle size on the bioavailability and bioactivity of perilla oil in rats. *Journal of Food Science*, *86*, 206-214. https://doi.org/10.1111/1750-3841.15537

Hwang, I.C., Kim, J., Kim, H.M., Kim, D.I., Kim, S.G., Kim, S.S. & Jang, C. (2009). Evaluation of *Toxicity of Plant Extract Made by Neem and Matrine against Main Pests and Natural Enemies*. *Korean Journal of Applied Entomology, 48*(1), 87-94.

Insecticide Resistance Action Committee (2019, Jan). *Brown planthopper.* https://irac-online.org/pests/nilaparvata-lugens/

Iqbal, N., Kumar, N., Agrawal, A. & Kumar, J. (2019). Development of highly stabilized neem oil microemulsion system: A green approach. *World Journal of Pharmaceutical Research, 8*(3),1507. 10.20959/wjpr20193-14435.

Irigaray, F.J.S., Fernando, M.J., Vicente, M., Ignacio, P.M. (2010). Acute and reproductive effects of Align®, an insecticide containing azadirachtin, on the grape berry moth, *Lobesia botrana*. *Journal of Insect Science*, *10*(1), 33. https://doi.org/10.1673/031.010.3301

Iyer, V., Cayatte, C., Guzman, B., Schneider-Ohrum, K., Matuszak, R., Snell, A., Rajani, G. M., McCarthy, M. P., & Muralidhara, B. (2015). Impact of formulation and particle size on stability and immunogenicity of oil-in-water emulsion adjuvants. *Human vaccines & immunotherapeutics, 11*(7), 1853–1864. https://doi.org/10.1080/21645515.2015.1046660

Jaiswal, M., Dudhe, R., & Sharma, P. K. (2015). Nanoemulsion: an advanced mode of drug delivery system. *Biotech*, *5*(2), 123–127. https://doi.org/10.1007/s13205-014-0214-0

Jing, S., Zhao, Y., Du, B., Chen, R. Lili, Z. & He, G. (2017). Genomics of interaction between the brown planthopper and rice. *Current Opinion in Insect Science*, *19*, 82-87. https://doi.org/10.1016/j.cois.2017.03.005.

Kala, S., Sogan, N., Agarwal, A., Naik, S.N., Patanjali, P.K. & Kumar, J. (2020). Biopesticides: Formulations and Delivery Techniques. In Chukwuebuka, E., & Barbara, S. (Eds). *Natural remedies for pest, disease and weed control* (pp. 209-220). Academic Press. https://doi.org/10.1016/B978-0-12-819304-4.00018-X.

Kathleen J.S. & Lin, S.Y. (2001). Dynamic surface tension and surfactant mass transfer kinetics: Measurement techniques and analysis. In Hari Singh Nalwa (Eds). *Handbook of surfaces and interfaces of materials* (pp. 55-60). Academic Press. ISBN 9780125139106. https://doi.org/10.1016/B978-012513910-6/50021-9.

Karise, R., Muljar, R., Smagghe, G., Kaart, T., Kuusik, A., Dreyersdorff, G., Williams, I.H. & Ma¨nd, M. (2015). Sublethal effects of kaolin and the biopesticides Prestop-Mix and BotaniGard on metabolic rate, water loss and longevity in bumble bees (*Bombus terrestris*). *Journal of Pest Science*, *89*(1). https://doi.org/10.1007/s10340-015-0649-z

Khan M.A. & Ahmad W. (2019). Synthetic Chemical Insecticides: Environmental and agro contaminants. In Khan M., Ahmad W. (Eds). *Microbes for Sustainable insect pest management. sustainability in plant and crop protection.* Springer, Cham. https://doi.org/10.1007/978-3-030-23045-6_1

Krishnaiah, N.V., Prasad, A.S., Rao, C.R., Pasalu, I.C., Lakshmi, J., Narayana, L., & Lingaiah, T. (2005). Effect of constant and variable temperatures on biological parameters of rice brown planthopper, *Nilaparvata lugens* (Stal). *Indian Journal of Plant Protection, 33*, 181-187.

Kohlmann, B., Arroyo, A., Macchi, P.A. & Palma, R. (2018). Biodiversity and Biomonitoring Indexes. In Britt, M., & Andrew, C. (Eds). *Integrated Analytical Approaches for Pesticide Management* (pp. 83-106). Academic Press. https://doi.org/10.1016/B978-0-12-816155-5.00006-3.

Kosman, E. & Cohen, Y. (1996). Procedures for calculating and differentiating synergism and antagonism in action of fungicide mixtures. *Phytopathology, 86*, 1263-1272.

Lakesh, M., Sukanta, K.P., Abinash, M., Debendra, N.B., Kailash, C.S., Pawan, K.A. & Manasi, D. (2021). Understanding brown planthopper resistance in rice: Genetics, biochemical and molecular breeding approaches. *Rice Science, 28*(6), 532-546. https://doi.org/10.1016/j.rsci.2021.05.013.

Latip, S.N.H.M. & Bakar, A.S.A. (2021). Life table and demographic parameters of brown planthopper (bph), *Nilaparvata lugens* (Homoptera: Delphacidae) on paddy for population studies. *Serangga, 26*(2), 216-225.

Lee, C.Y. (2000). Sublethal effects of insecticides on longevity, fecundity and behaviour of insect pests: A review. *Journal of Bioscience, 11*(1-2).

Lengai, G.M.W., Muthomi, J.W. & Mbega, E.R. (2020). Phytochemical activity and role of botanical pesticides in pest management for sustainable agricultural crop production. *Scientific African, 7.* https://doi.org/10.1016/j.sciaf.2019.e00239.

Levchenko, M. & Silivanova, E. (2019). Synergistic and antagonistic effects of insecticide binary mixtures against house flies (*Musca domestica*). *Regulatory Mechanisms in Biosystems, 10,* 75-82. 10.15421/021912.

Liu, S., Luo, J., Liu, R., Zhang, C., Duan, D., Chen, H., Bei, W. & Tang, J. (2018). Identification of *Nilaparvata lugens* and its two sibling species (*N. bakeri* and *N. muiri*) by direct multiplex PCR. *Journal of economic entomology*, 111. https://doi.org/10.1093/jee/toy232.

Lu, G.W. & Gao, P. (2010). Emulsions and microemulsions for topical and transdermal drug delivery. In Vitthal S.K (Eds). *Personal Care & Cosmetic Technology. Handbook of Non-Invasive Drug Delivery Systems* (pp. 59-94). William Andrew Publishing. pp59-94. https://doi.org/10.1016/B978-0-8155-2025-2.10003-4.

Maestroni, B., Ferris, I.G., Correll, R., Kohlmann, B., Nordgaard, A., Kookana, R.S. & Cannavan, A. (2018). Generic guidelines on integrated analytical approaches to assess indicators of pesticide management practices at a catchment scale: Black-box monitoring and the laboratory's role in fostering good agricultural practice. In Britt, M., & Andrew, C. (Eds). *Integrated analytical approaches for pesticide management* (pp. 7-27). Academic Press. https://doi.org/10.1016/B978-0-12-816155-5.00002-6.

Maizatul, I & Nor Farahiya, G. (2018). Phytochemical and pharmacological profile of Kaduk (*Piper sarmentosum* Roxb.). *Malayan Nature Journal, 70*(2), 195-202.

Malaysia Biodiversity Information System (MyBIS). *Piper sarmentosum*. Accessed via https://www.mybis.gov.my/sp/44223. [Retrieved 18 February 2022].

Maneerat, J., Suchada, P. & Pornsak, S. (2013). Effect of pH on stability of oil-in-water emulsions stabilized by Pectin-Zein complexes. *Advanced Materials Research, 747*, 127-130. 10.4028/www.scientific.net/AMR.747.127.

Marco, A.F, Roberto, C.O.., Jorge, N.C. & Krishnaswamy, R. (2005). Viscosity of water-in-oil emulsions: Variation with temperature and water volume fraction. *Journal of Petroleum Science and Engineering*, *48*(3–4), 169-184. https://doi.org/10.1016/j.petrol.2005.06.014.

María Vita,P.M., Alejandro, A.M., Emilio, M.P., Ramón, S.S. & Elvia María, P.L. (2004). Influence of the speed mixing-on viscosity and droplet size of oil in water emulsions, *Petroleum Science and Technology*, *22*(7-8), 1035-1043, https://doi.org/10.1081/LFT-120038709

Marziyeh, C, Dzolkhifli, O., Mahiran, B. & Norhayu, A. (2017). Preparation and characterization of neem oil nanoemulsion formulations against *Sitophilus oryzae* and *Tribolium castaneum* adults. *Journal of pesticide science*, *42*(4), 158–165. https://doi.org/10.1584/jpestics.D17-032

Matharu, K., & Tanwar, P.S. (2020). Efficacy of different insecticides against brown planthopper, *Nilaparvata lugens* (Stål) in rice. *International Journal of Chemical Sciences*, *8*(3), 870-873. 10.22271/chemi.2020.v8.i3k.9310.

McAllister, E.W. (2014). Measurement. In McAllister, E.W. (Eds). *Pipeline rules of thumb handbook (Eighth Edition)*. (pp. 541-624). Gulf Professional Publishing. ISBN 9780123876935. https://doi.org/10.1016/B978-0-12-387693-5.00015-2.

Meshram, S., Bisht, S. & Gogoi, R. (2022). Current development, application and constraints of biopesticides in plant disease management. *Biopesticides*, *2*(14), 207-224. https://doi.org/10.1016/B978-0-12-823355-9.00004-3.

Michel, C., Armelle, R. & Virginie, L. (2013). Molten carbonates from fuel cells to new energy devices. In Frédéric Lantelme & Henri Groult (Eds). *Molten salts chemistry* (pp. 355-371). Elsevier. ISBN 9780123985385. https://doi.org/10.1016/B978-0-12-398538-5.00017-2.

Min, S., Lee, S.W., Choi, B.R., Lee, S.H & Kwon, D.H. (2014). Insecticide resistance monitoring and correlation analysis to select appropriate insecticides against *Nilaparvata lugens* (Stål), a migratory pest in Korea. *Journal of Asia-Pacific Entomology*, *17*(4), 711-716.

Alzohairy M. A. (2016). Therapeutics role of *Azadirachta indica* (Neem) and their active constituents in diseases prevention and treatment. *Evidence-based complementary and alternative medicine : eCAM, 2016*, 7382506. https://doi.org/10.1155/2016/7382506

Moldoveanu, S. & David, V. (2021). Solvent extraction. In Serban, M., & Victor, D. (Eds). *Modern sample preparation for chromatography (Second Edition).* (pp. 191-279). Elsevier. https://doi.org/10.1016/B978-0-12-821405-3.00015-0.

Mondal, D. & Tamal, M. (2012). A Review on efficacy of *Azadirachta indica* A. Juss based biopesticides: An Indian perspective. *Research Journal of Recent Sciences, 1*(3), 2277-2502. 1. 2277-2502.

Mordue, A.J., & Nisbet, A.J. (2000). Azadirachtin from the neem tree Azadirachta indica: Its action against insects. *Anais da Sociedade Entomológica do Brasil, 29*(4), 615-632. https://doi.org/10.1590/S0301-80592000000400001

Muduli, L., Muduli, Pradhan, S.K., Mishra, A., Bastia, D.N., Samal, K.C., Agrawal, P.K., & Dash. M. (2021). Understanding brown planthopper resistance in rice: Genetics, biochemical and molecular breeding approaches. *Rice science, 28*, 532-546. https://doi.org/10.1016/j.rsci.2021.05.013

Muhammad Aufa M.B., Mohd Fitri M., Nuraini A.A.S, Arina, M.N., Nurul Syakira, S., Badril Hisham, A.B. & Mohd Khusairy, K. (2021). Evaluation of Light Trap System in Monitoring of Rice Pests, Brown Planthopper (*Nilaparvata lugens*). *Advances in Agricultural Food Research Journal, 2*(2), a0000187. https://doi.org/10.36877/aafrj.a0000187

Nathan, S.S., Choi, M.Y., Paik, C.H., Seo, H.Y., Kim, J.D., & Kang, S.M. (2007). The toxic effects of neem extract and azadirachtin on the brown planthopper, *Nilaparvata lugens* (Stål) (BPH) (Homoptera: Delphacidae). *Chemosphere, 67*(1), 80-88. https://doi.org/10.1016/j.chemosphere.2006.09.045.

National Center for Biotechnology Information. (2022). PubChem Compound Summary for CID 5281303, *Azadirachtin*. Retrieved February 17, 2022 from https://pubchem.ncbi.nlm.nih.gov/compound/Azadirachtin.

National Center for Biotechnology Information. (2022). PubChem Compound Summary for CID 6440616, (2E,4E)-1-(Pyrrolidin-1-yl)deca-2,4-dien-1-one. Retrieved February 17, 2022 from https://pubchem.ncbi.nlm.nih.gov/compound/2E_4E_-1-pyrrolidin-1-yldeca-2_4-dien-1-one.

National Center for Biotechnology Information. (2022). PubChem Compound Summary for CID 6438710, *Sarmentosine*. Retrieved February 17, 2022 from https://pubchem.ncbi.nlm.nih.gov/compound/Sarmentosine.

Ng, L.T.,Yuen, P.M., Loke, W.H. & Azizol, A.K. (2003). Effects of *Azadirachta excelsa* on feeding behaviour, body weight and mortality of *Crocidolomia binotalis* Zeller (Lepidoptera: Pyralidae). *Journal of the Science Food and Agriculture*, *83*, 1327. https://doi.org/10.1002/jsfa.1542

Niknam, S.M., Escudero, I. & Benito, J.M. (2020). Formulation and preparation of water-in-oil-in-water emulsions loaded with a phenolic-rich inner aqueous phase by application of high energy emulsification methods. *Food*, *9*, 1411. https://doi.org/10.3390/foods9101411

Norliyana Farzana, Z., Wan Azlina, W.A.K., Salmiaton, A., Norhayu, A. & Yamuna, C. (2021). Development of bio-pesticides against *Metisa plana* walker bagworm (Lepidoptera: Psychidae). *Journal of Oil Palm, Environment & Health*, *12*, 109-117. https://doi.org/10.5366/jope.2021.08

Odugbemi, T. (2008). *A textbook of medicinal plants from Nigeria*. Tolu Odugbemi.

Ohkawa, H., Miyagawa, H., & Lee, P. (2007). *Pesticide chemistry: Crop protection, public health, environmental safety.* John Wiley & Sons. https://doi.org/10.1002/9783527611249.

Omar, D., Asib, N., Choupanian, M., & Kamarudin, N. (2016). Nanoemulsion Formulation of Botanical Insecticides for Effective Insect Control.

Otto A. & du Plessis J. (2015). The effects of emulsifiers and emulsion formulation types on dermal and transdermal drug delivery. In Dragicevic N., & Maibach H. (eds) *Percutaneous penetration enhancers chemical methods in penetration enhancement*. Springer. https://doi.org/10.1007/978-3-662-45013-0_16

Pariyani, R., Ismail, I.S., Ahmad Azam, A., Abas, F., Shaari, K. & Sulaiman, M.R. (2015). Phytochemical screening and acute oral toxicity study of java tea leaf extracts. *BioMed Research International*, *8*. https://doi.org/10.1155/2015/742420

Parrotta, J.A. (2001). Healing plants of Peninsular India. CAB International. https://doi.org/10.1079/9780851995014.0000

Pezzani, R., Salehi, B., Vitalini, S., Iriti, M., Zuñiga, F. A., Sharifi-Rad, J., Martorell, M., & Martins, N. (2019). Synergistic effects of plant derivatives and conventional chemotherapeutic agents: An update on the cancer perspective. *Medicina, 55*(4), 110. https://doi.org/10.3390/medicina55040110

Piplani, M., Bhagwat, D.P., Singhvi, G., Sankaranarayanan, M., Balana-Fouce, R., Vats, T. & Chander, S. (2019). Plant-based larvicidal agents: An overview from 2000 to 2018. *Experimental Parasitology, 199*, 92-103. https://doi.org/10.1016/j.exppara.2019.02.014.

Piyaphongkul, J., Pritchard, J. & Bale, J. (2012). Heat stress impedes development and lowers fecundity of the brown planthopper *Nilaparvata lugens* (Stål). *PLoS ONE, 7*(10), e47413. https://doi.org/10.1371/journal.pone.0047413

Prasad, D. (2007). *Sustainable pests management*. Daya.

PROSEA Foundation, 2022. *Azadirachta indica*. In: Invasive Species Compendium. Wallingford, UK: CAB International. www.cabi.org/isc. Retrieved February 18, 2022 from https://www.cabi.org/isc/datasheet/8112#toPictures

Purba, R.A.P., Paengkoum, S. & Paengkoum, P. (2021). Development of a simple high-performance liquid chromatography-based method to quantify synergistic compounds and their composition in dried leaf extracts of *Piper sarmentosum* Roxb. *Separations*, *8*(9),152. https://doi.org/10.3390/separations8090152

Qin, W., Huang, S., Li, C., Chen, S. & Peng, Z. (2010). Biological activity of the essential oil from the leaves of *Piper sarmentosum* Roxb. (Piperaceae) and its chemical constituents on *Brontispa longissima* (Gestro) (Coleoptera: Hispidae). *Pesticide Biochemistry and Physiology*, *96*, 132-139. 10.1016/j.pestbp.2009.10.006.

Qurban, A. & Asad, A. (2020). Effect of insect growth regulators on fecundity, fertility and adult emergence of *Tribolium castaneum* (herbst) and *Trogoderma granarium* (everts). *Pakistan Journal of Agricultural Sciences*, *57,* 799-805. 10.21162/PAKJAS/20.198.

Rafferty, J.P. (2022). *r-selected species. Encyclopedia Britannica*. https://www.britannica.com/science/r-selected-species

Raj, J., Chandra, M., Dogra, T. D., Pahuja, M., & Raina, A. (2013). Determination of median lethal dose of combination of endosulfan and cypermethrin in wistar rat. *Toxicology international, 20*(1), 1–5. https://doi.org/10.4103/0971-6580.111531

Ravera, F., Dziza, K., Santini, E., Cristofolini, L. & Liggieri, L. (2021). Emulsification and emulsion stability: The role of the interfacial properties. *Advances in Colloid and Interface Science, 288*, 102344. https://doi.org/10.1016/j.cis.2020.102344.

Rawani, A., Ghosh, A. & Chandra, G. (2010). Mosquito larvicidal activities of *Solanum nigrum* L. leaf extract against *Culex quinquefasciatus* Say. *Parasitol Research, 107*(5), 1235-1240. https://doi.org/10.1007/s00436-010-1993-9

Rechcigh, J.E and Rechcigh, N.A. (2000). *Biological and biotechnological control of insect pest*. Lewis Publishers. New York.

Rizzo, E. & Bazzoli, N. (2020). Reproduction and embryogenesis. In Bernardo, B., Elisabeth, C.U. (Eds). *Cyrino. biology and physiology of freshwater neotropical fish*. (pp. 287-313). Academic Press. https://doi.org/10.1016/B978-0-12-815872-2.00013-0.

Roell, K.R., Reif, D.M. & Motsinger-Reif, A.A. (2017). An introduction to terminology and methodology of chemical synergy—Perspectives from across disciplines. *Frontiers in Pharmacology, 8*. DOI=10.3389/fphar.2017.00158

Saini, A., Saini, G., Singh, B., Vyas, M., Verma, S. & Prakash, O. (2019). Synergistic effect of *Azadirachta indica* and *Curcuma longa* with fluconazole gel against *Candida albicans*. *International Journal of Pharmaceutical Sciences and Research, 10*(2), 692-700.

Sakine, U.K. (2012). Insecticide resistance, insecticides - Advances, In Dr. Farzana Perveen (Eds), *Integrated Pest Management*. InTech ISBN: 978-953-307-780-2, , Available from: http://www.intechopen.com/books/insecticides-advances-in-integrated-pest-management/insecticideresistance

Sankara, R.K., Raja, K.S., Deepak, K., Arun, S.R., & Gopalakrishna, B.K. (2019). Flora of Peninsular India. http://peninsula.ces.iisc.ac.in/plants.php?name=*Azadirachta indica*. Downloaded on 5 August 2022.

Sara, P., Nicholas, C., John, T., Ken, K. & Graeme, I.P. (2000). Biosensing the acute toxicity of metal interactions: are they additive, synergistic, or antagonistic? *Environmental Toxicology and Chemistry, 19*(3), 775–780.

Satti, A.A., Elamin, M.M., &, Mohamed, A.E. (2010). Insecticidal activities of neem (*Azadirachta indica* A. Juss) seeds under laboratory and field conditions as affected by different storage durations. *Agriculture and Biology Journal of North America, 1,* 1001-1008. 10.5251/abjna.2010.1.5.1001.1008.

Satti, A.A., Elamin, M.M., & Futuwi, A.I. (2013). Insecticidal effects of neem (*Azadirachta indica* A. Juss) oils obtained from neem berries stored at different periods. *The Experiment, 6*, 330-337.

Schäpers, A., Petrén, H., Wheat, C. W., Wiklund, C., & Friberg, M. (2017). Female fecundity variation affects reproducibility of experiments on host plant preference and acceptance in a phytophagous insect. Proceedings. *Biological sciences*, *284*(1849), 20162643. https://doi.org/10.1098/rspb.2016.2643

Seriana, I., Akmal, M., Darusman, D., Wahyuni, S., Khairan, K., & Sugito, S. (2021). Neem leaf (*Azadirachta indica* A. Juss) ethanolic extract on the liver and kidney function of rats. *The Scientific World Journal, 2021,* 7970424. https://doi.org/10.1155/2021/7970424

Sharifah Farhana, S.A.R, Kamaruzaman, S & Dzolkifli, O. 2016. *Piper sarmentosum* Roxb.: A mini review of ethnobotany, phytochemistry and pharmacology. *Journal of Analytical and Pharmaceutical Research, 2*(5), 00031. https://doi.org/10.15406/japlr.2016.02.00031.

Sharma, R., Kumari, A., Singh, N.S., Singh, M.K., Dubey, S., Iqbal, N. & Patanjali, P.K. (2019). Development and stability enhancement of neem oil based microemulsion formulation using botanical synergist. *Journal of Molecular Liquids*, *296*, 112012. https://doi.org/10.1016/j.molliq.2019.112012.

Siti Efliza, A., Norazlinaliza, S., Norhayu, A. Dzolkhifli, O. & Gwenoline, E.C.L. (2019). Optimization and Characterization of Palm Oil-based Nanoemulsion Loaded with *Parthenium hysterophorus* Crude Extract for Natural Herbicide Formulation. *Journal of Oleo Science, 68*(8), 747-757

Siti Fairuz, Y., Farah Farhana, H., Norhayu, A., Mahmud, T.M.M. & Siti Izera, I. (2021). Development of *Vernonia amygdalina* leaf extract emulsion formulations in controlling gray mold disease on tomato (*Lycopersicon esculentum* Mill.). *Agronomy*, *11*(2), 373. https://doi.org/10.3390/agronomy11020373

Siti Marjiana, I., Chua, K.H., Amilia, A and Azizah, U. (2018). Piper sarmentosum as an antioxidant: A systematic review. *Sains Malaysiana Journa,l 47*(10).

Solanke, K. R., & Yadav, S. N. (2019). Study of physical and electrical properties of selected biopesticides to characterize spray. J*ournal of Biopesticide International*, *15*(2), 105-111.

Sonal, D. & Pankaj, K. (2014). *Azadirachta indica*: A plant with versatile potential. RGUHS *Journal of Pharmaceutical Sciences, 4*, 39-46.

Sonam, K.S. & Guleria, S. 2017. Synergistic antioxidant activity of natural products. *Ann Pharmacol Pharm, 2*(8), 1086.

Sowmiya. C. & Sankarganesh, E. (2019). Pesticide resistance and its management in insect pests. *Agrobios Newletter, 18*(6), 82-83.

Sporleder, M. & Lacey, L. (2013). Biopesticides. In Andrei Alyokhin, Charles Vincent, Philippe Giordanengo (Eds). *Insect pests of potato*. (pp. 463-497). Academic Press. https://doi.org/10.1016/B978-0-12-386895-4.00016-8.

Sri Yulianto, P., Subanar, S. & Daryono, B. (2013). The prediction of population dynamics based on the spatial distribution pattern of brown planthopper (*Nilaparvata lugen* Stal.) using exponential smoothing –

Local spatial statistics. *Journal of Agricultural Science*, 5. 10.5539/jas.v5n5p209.

Srinivas, M., Varma, N.R.G., Devi, R.S., & Jagadeeshwar, R. (2016). Effect of temperature on rice brown plant hopper (Nilaparvata lugens Stall). *Journal of Research Professor Jayashankar Telangana State Agricultural University, 44*(1/2), 114-117.

Stoner, K. (n.d). *Approaches to the biological control of insect pests*. The Connecticut Agricultural Experiment Station. https://portal.ct.gov/CAES/Fact-Sheets/Entomology/Approaches-to-the-Biological-Control-of-Insect-Pests#:~:text=Summary-,Biological%20control%20is%20the%20use%20of%20living%20organisms%20to%20suppress,predators%2C%20parasitoids%2C%20and%20pathogens.

Suiter, D. R. & Scharf, M. E. (2015). Insecticide basics for the pest management professional. *UGA Bulletin 1352*.

Szymon, C.,, Milena,k., Paweł, M., & Grzegorz, R.(2014). Synthetic insecticides – is there an alternative?. *Polish Journal of Environmental Studies, 23*(2), 291-302.

Tadros, T. (2013). Ostwald ripening. In Tadros T. (eds) *Encyclopedia of Colloid and Interface Science*. Springer. https://doi.org/10.1007/978-3-642-20665-8_124

Tallarida, R. J. (2011). Quantitative methods for assessing drug synergism. *Genes & cancer*, *2*(11), 1003–1008. https://doi.org/10.1177/1947601912440575

Tamilvanan, S., Raja, S.T, & Ruchi, S. (2017). Multifunctional nanosized emulsions for theragnosis of life threatening diseases. IN Ecaterina Andronescu, Alexandru Mihai Grumezescu (eds). *Micro and nano technologies, nanostructures for drug delivery.* (pp. 579-617). Elsevier. https://doi.org/10.1016/B978-0-323-46143-6.00019-1.

Tang, M., Lv, L., Jing, S., Zhu, L. & He, G. (2010). Bacterial Symbionts of the brown planthopper, *Nilaparvata lugens* (Homoptera: Delphacidae). *Applied and Environmental Microbiology*, 1740-1745.

Tarapore, D., Floreano, D. & Keller, L. (2010). Task-dependent influence of genetic architecture and mating frequency on division of labour in social insect societies. *Behavioral Ecology and Sociobiology, 64*(4), 675–684.

Tembo, Y., Mkindi, A. G., Mkenda, P. A., Mpumi, N., Mwanauta, R., Stevenson, P. C., Ndakidemi, P. A., & Belmain, S. R. (2018). Pesticidal plant extracts improve yield and reduce insect pests on legume crops without harming beneficial arthropods. *Frontiers*, *9*, 1425. https://doi.org/10.3389/fpls.2018.01425

Thilak, M., Haiou, Q., Desiree, V.H., Siyam, M.A., Angel, P., Taylor, I. (2019). Characterization of nanomaterials: Tools and challenges. In Amparo

López Rubio, Maria José Fabra Rovira, Marta martínez Sanz, Laura Gómez Gómez-Mascaraque (eds). *Micro and nano technologies. Nanomaterials for food applications*. (pp. 313-353). Elsevier. ISBN 9780128141304. https://doi.org/10.1016/B978-0-12-814130-4.00011-7.

Ting, D., Tomohisa, N.., Hideyuki, N., Qui, T.C.M. (2020). Particle size distribution analysis of oil-in-water emulsions using static and dynamic ultrasound scattering techniques. *Ultrasonics*, *108,* 106117. https://doi.org/10.1016/j.ultras.2020.106117.

Ugwu, J.A. (2020). Insecticidal activity of some botanical extracts against legume flower thrips and legume pod borer on cowpea *Vigna unguiculata* L. Walp. *The Journal of Basic and Applied Zoology*, *81*(13). https://doi.org/10.1186/s41936-020-00153-3

Vakil, V. & Trappe, W. (2019). Drug combinations: Mathematical modeling and networking methods. *Pharmaceutics, 11*(5), 208. https://doi.org/10.3390/pharmaceutics11050208

Verma, P., Kamboj, P., Ranjan, S. & Mahesh K. (2010). anti-inflammatory and analgesic activity of aqueous extract of *Trichosanthes bracteata* fruits in animal model. *Pharmacologyonline, 1,* 8-16.

Vijayakumar, S. (2019). Stability studies on nanomaterials used in drugs, in Shyam S. Mohapatra, Shivendu Ranjan, Nandita Dasgupta, Raghvendra Kumar Mishra, Sabu Thomas (Eds). *Micro and nano technologies, characterization and biology of nanomaterials for drug delivery* (pp. 425-444). Elsevier. https://doi.org/10.1016/B978-0-12-814031-4.00015-5.

Wafaa, M.H., Rowida, S.B. & Hussein, A.H.S.A.A. (2017) | Karol Ujházy (Reviewing Editor) Botanical insecticide as simple extractives for pest control. *Cogent Biology*, *3*, 1. https://doi.org/ 10.1080/23312025.2017.1404274

Wang, Y., Chen, J.,Zhu, Y.C., Ma, C., Huang, Y., & Shen, J. (2008). Susceptibility to neonicotinoids and risk of resistance development in the brown planthopper, *Nilaparvata lugens* (Stal) (Homoptera: Delphacidae). *Pest management science, 64*, 1278-84. https://doi.org/ 10.1002/ps.1629.

Wang, Y., Lv, L., Yu, Y., Yang, G., Xu, Z., Wang, Q. & Cai, L. (2017). Single and joint toxic effects of five selected pesticides on the early life stages of zebrafish (*Denio rerio*). *Chemosphere, 170*, 61-67. https://doi.org/10.1016/j.chemosphere.2016.12.025.

Watters, R.J., Kester, M., Tran, M.A., Loughran, T. P. & Xin, L. (2012). Development and use of ceramide nanoliposomes in cancer, in Nejat Düzgüneş (Eds). *Methods in enzymology* (pp. 89-108). Academic Press. https://doi.org/10.1016/B978-0-12-391860-4.00005-7.

Waxham, M.N. (2007). Molecular mobility in cells examined with optical methods. In Andrew J. Bean (Eds). *Protein trafficking in neurons* (pp.

3-27). Academic Press. https://doi.org/10.1016/B978-012369437-9/50003-7.

Weiquan, Q., Shanchun, H., Chaoxu, L., Siting, C. & Zhengqiang, P. (2010). Biological activity of the essential oil from the leaves of *Piper sarmentosum* Roxb. (Piperaceae) and its chemical constituents on *Brontispa longissima* (Gestro) (Coleoptera: Hispidae). *Pesticide Biochemistry and Physiology*, *96*(3), 132-139. https://doi.org/10.1016/j.pestbp.2009.10.006.

Wiart, C. (2006). *Medicinal plants of Asia and the Pacific*. Taylor and Francis.

Winand, H. (2006). *Pesticide Safety Fact Sheet: Toxicity of Pesticides*. Publications Distribution Center, The Pennsylvania State University.

Xu, H.J & Zhang, C. X. (2017). Insulin receptors and wing dimorphism in rice planthoppers. *Philosophical Transactions of the Royal Society*, *372*(1713), 20150489. http://doi.org/10.1098/rstb.2015.0489

Yamashita, Y., Miyahara, R. & Sakamoto, K. (2017). Emulsion and emulsification technology. In Kazutami Sakamoto, Robert Y. Lochhead, Howard I. Maibach, Yuji Yamashita (Eds). *Cosmetic Science and Technology* (pp. 489-506). Elsevier. https://doi.org/10.1016/B978-0-12-802005-0.00028-8.

Yang, H., Dong, J., Hu, Z., Li, D., Fang, F. & Zhai, B. (2019). Temporary inhibition of positive phototaxis in emigratory population of *Nilaparvata lugens* by mark-release-recapture. *PLoS ONE, 14*(9), e0222214. https://doi.org/10.1371/journal.pone.0222214

Zainuddin, N. J., Ashari, S. E., Salim, N., Asib, N., Omar, D., & Lian, G. (2019). Optimization and characterization of palm oil-based nanoemulsion loaded with *Parthenium hysterophorus* crude extract for natural herbicide formulation. *Journal of oleo science, 68*(8), 747–757. https://doi.org/10.5650/jos.ess18209

Zarkani, A., & Prijono, D. (2009). Insecticidal effectiveness of *Piper retrofractum* and *Tephrosia vogelii* extracts against *Crocidolomia pavonana* and *Plutella xylostella* and the safety of the extracts to *Dialegma semiclausum*. *Journal of ISSAAS [International Society for Southeast Asian Agricultural Sciences, 15*(1), 209-210.

Zhang, Z.X. (2016). Effect of temperature on rock fracture. In Zong-Xian Zhang (Eds). *Rock fracture and blasting* (pp. 111-133). Butterworth-Heinemann. ISBN 9780128026885. https://doi.org/10.1016/B978-0-12-802688-5.00005-1.

Zhu K.Y. (2004). *Insecticide Toxicity*. In: Encyclopedia of Entomology. Springer, Dordrecht. https://doi.org/10.1007/0-306-48380-7_2190